

Pressure filtration and slip casting of mixed alumina–zircon suspensions

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Abstract

Mullite-zirconia based ceramics can be obtained from zircon raw material and alumina. Rheological properties of aqueous 48 vol.% suspensions containing a mixture of zircon and α -alumina powders were determined to obtain the optimum dispersion conditions for colloidal processing of dense mullite-zirconia composites. Powder compositions with different alumina/zircon (wt.%) ratios in the mixtures were studied. Colloid stability as a function of pH for zircon and alumina, with and without polyacrylate, was studied by sedimentation tests. The effect of the chemical composition of the mixed powders and addition of polyacrylate on the viscosity of the suspensions were examined. Green density of compacts that were consolidated by slip casting and pressure filtration at 8 MPa were correlated with rheological properties of the suspensions and their compositions. Pressure filtered bodies were slightly less dense than the compacts prepared by slip casting and the dried relative density increased with increasing the degree of dispersion of the particles. After sintering at 1600°C for 2 h, the compacts from well-dispersed suspensions reached high density showing a fine and homogeneous microstructure. © 2001 Elsevier Science Ltd. All rights reserved.

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1. Introduction

The preparation of mullite-zirconia composites using the reaction sintering (RS) method has attracted the attention of the scientists since good-quality zircon is readily available from beach sand and a cheaper price, thus making the development cost effective.¹ This method consists of the reaction of zircon with alumina at 1600–1700°C, to produce 3Al₂O₃·2SiO₂ and ZrO₂.

The mechanism of mullite-zirconia formation was earlier described in detail.^{2,3}

Although zircon are successfully used to produce a mullite-zirconia composite by conventional dry preparation methods, there are few available works on the casting behavior of commercial zircon powder using an organic surfactant/polyelectrolyte.^{4,5} It is widely recognized that the degree of dispersion of the particles greatly influences the flow properties of concentrated ceramic suspensions which are related to the green and sintered properties of the product.

Polyacrylate based dispersants are commonly used to improve and control the colloidal stability and rheological properties of concentrated ceramic suspension.^{6–8} Colloidal stability of concentrated suspensions using polyelectrolytes was attributed to both electrostatic repulsion and steric stabilization mechanisms. Adsorption of anionic polyelectrolytes on the powder surface increase the negative surface charge and produce some steric repulsion.

Effective stabilization by adsorption of polyelectrolytes from aqueous solutions requires strongly bounded polyelectrolytes and higher surface coverage. Therefore, is important to know more about the affinity of the polyelectrolyte for the powder surface as well as the effects of the presence of excessive amounts of dispersant in solution on rheological behavior and on colloidal processing of the concentrated suspensions.

In this work, the influence of the chemical composition, particle size distribution of the mixture and polyacrylate content on the viscosity of 48 vol.% alumina-zircon suspensions was examined. Suspension stability was evaluated by rheological measurements and correlated with the adsorbed amount of polyacrylate on the

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mixture. Characteristics of dried and sintered compacts at 1450 and 1600°C for 2 h obtained by slip casting and pressure filtration such as density, phase composition and microstructure were determined.

2. Materials and methods

2.1. Materials

Alumina powder used is a fine and pure α -Al₂O₃ (A-16SG, Alcoa Inc., USA) and contains Na₂O and MgO as the main impurities.^{9,10} The mean particle size determined was 0.4 μ m and the specific surface area (BET) 9.5 m²/g.¹¹ The isoelectric point has been determined to be 9.¹¹

From the chemical analysis results the zircon powder used (Mahlwerke Kreutz, Mikron, Germany) contains minor amounts of Al₂O₃: 0.9%, TiO₂: 0.11%, Fe₂O₃: 0.05%, P₂O₅: 0.1% and HfO₂: 1.3%.

The mean particle size determined was 2 μ m (Sedigraph 5000D, Micromeritics) and the specific surface area (BET) 4.1 m²/g. The isoelectric point (pH_{iep} = 5) was determined through the electroacoustic technique (ESA 8000, Matec Instruments) on a 1 vol.% suspension.¹²

2.2. Suspension preparation and characterization

Alumina–zircon mixtures of different proportions: stoichiometric (theoretical composition), with an excess of alumina, and with a zircon excess were prepared. The alumina/zircon (wt./wt.%) ratios of the mixtures were: 45.5/54.5, 51.7/48.3 and 35.1/64.9 that correspond to the following alumina/zircon molar ratios of 1.5 (stoichiometric 3:2), 1.9 and 0.95, respectively. Aqueous 48 vol.% suspensions at pH 9.1–9.2 were prepared by adding the powder to aqueous solutions with different polyacrylate content (Dolapix CE64, Zschimmers and Schwartz) and NH₄OH. After mixing, the suspensions were ultrasonicated for 30 min. Flow curves of the suspensions were obtained using a rotational viscometer Haake vt550 of coaxial cylinders with NV measure system at 25°C.

2.3. Polyacrylate adsorption measurements

According to Baklouti et al.⁸ a gravimetric technique can be used to give a semiquantitative measure of the adsorption of polyelectrolyte on alumina. Thus, the adsorbed amount of polyacrylate on the mixed powder was determined using a Netzsch STA 409 thermo-balance at a heating rate of 10°C/min.

Suspensions were prepared at pH near 9 (9.1–9.2) so that the adsorbed amount can be determined at constant pH. After suspension centrifugation and washing, the solid was dried at 60°C and the weight loss between 200 and 600°C was determined. This weight loss was

used to calculate the relative amount of polymer adsorbed on the mixtures.

2.4. Slip casting, pressure filtration and characterization of the compact

The consolidation of suspensions by slip casting was carried out in a plaster mould to produce samples in a form of bars (12×12×60 mm³).

Pressure filtration was done on a metallic sintered porous filter with a wet filter paper placed on it, uniaxial pressure was employed. The pressure of 8 MPa was applied from the top and the liquid moved through the porous filter. Pressure was maintained constant for 30 min. Cylindrical cast samples (25 mm diameter and 10–15 mm thick) were obtained.

Bars and cylindrical compacts were dried 24 h at room temperature and then at 110°C up to a constant weight. Relative density (% of the theoretical) was measured by Hg immersion.

After sintering (1450 and 1600°C for 2 h), density was determined by the water absorption method. Fired bodies were characterized by XRD (Phillips 3020 goniometer with PW3710 controller, Cu-K α radiation and Ni filter at 40 kV–20 mA). Component segregation and microstructure was observed by SEM (Phillips 505-Scanning electron microscope with EDAX accessory).

3. Results and discussion

3.1. Colloidal stability of diluted suspensions

3.1.1. Effect of pH

The isoelectric point (iep) of alumina used has been determined to be near 9 and the zeta potential became slightly negative with further increase in pH.¹¹ Depending on the pH, the association–dissociation reaction of H⁺ with Al–OH surface group develops AlOH₂⁺ or AlO[–] charged sites.

Zeta potential vs pH curve of zircon indicated that the iep is 5.¹² The magnitude of the negative zeta potential strongly increased with increasing pH from the pH_{iep}. Thus the electrostatic interaction between zircon and alumina particles is attractive between pH 5 and 9.

Particle size distribution measurements were used to determine the colloidal stability of diluted suspensions. Well dispersed particles give small average particle size d₅₀ because they settle separately. Whereas big aggregates resulting from flocculation of the individual particles show a higher d₅₀ values than that of the well dispersed suspension.

Fig. 1 (curves a and b) shows the average particle size d₅₀ as a function of pH for alumina and zircon, respectively. For the zircon suspension, high d₅₀ values of 12–13 μ m were observed at pH close to 5 due to a

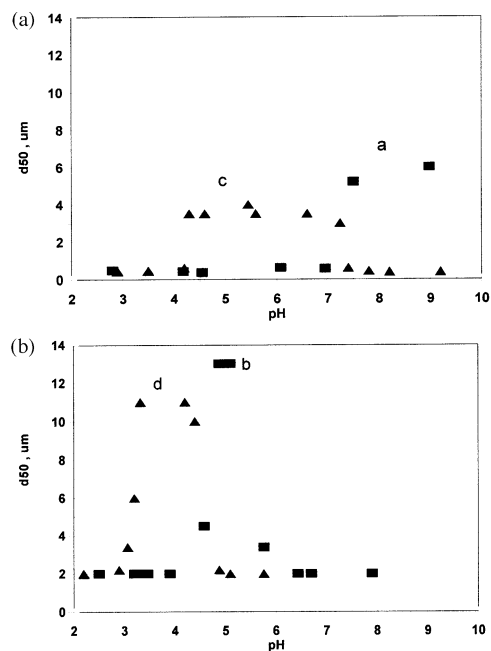


Fig. 1. Average particle size d_{50} for zircon and alumina powders as a function of pH with and without polyacrylate; a: alumina, b: zircon, c: alumina with 0.44 wt.% of polyacrylate, d: zircon with 0.05 wt.% of polyacrylate.

poor dispersion of zircon particles in agreement with the determined iep value from electroacoustic technique.¹² In a narrow range of pH, below or above pH_{iep} , d_{50} values decreased to 2 μm indicating high electrostatic repulsion between particles. For alumina suspension d_{50} gradually increased from 0.4 to 6 μm by changing the pH of 4 to 9 (pH_{iep}).

Colloidal stability of suspensions at pH 9 are related to zeta potential values as curves a and b in Fig. 1 indicated a stable suspension of zircon having a high negative zeta potentials (-80 mV) whereas in the alumina suspension particles exhibiting a neutral or slight negatively charged surface (-3 mV) were flocculated.

3.1.2. Effect of polyacrylate additions

The d_{50} values as a function of pH for alumina and zircon powders [Fig. 1 (curves c and d)] indicated that the degree of dispersion of the alumina and zircon suspension at the pH_{siep} improved with addition of polyacrylate as lower d_{50} values of 0.4 and 2 μm , respectively, were obtained. Based on zeta potential vs. pH measurements for the powders containing polyacrylate,¹² stabilization of the suspensions was provided by adsorbed polyacrylate anion on both powders that generated higher negative zeta potentials thereby increasing electrostatic repulsion between particles.

For the alumina suspension in a pH range from 9 to 7 little effect of the pH on the d_{50} values was found. At pH 7, d_{50} increased to 4 μm indicating a coagulated suspension due to negative charges of the coated alumina

were neutralized by the presence of more positive surface AlOH_2^+ groups at low pH. Also, the degree of dissociation of polyacrylate is smaller at pH 7 than that in basic conditions (pH 9).⁷ Further decrease in pH to 4 redispersed the particles as d_{50} decreased to 0.4 μm . This result may be explained by a charge reverse mechanism due to an excess of positive charges.

Because of the small specific surface area, an added polyacrylate concentration as low as 0.05 wt.% improved the degree of dispersion of the zircon. At pH 4.4–3.5, d_{50} values increased to 11 μm in concordance with the pH_{iep} of the polyacrylate coated zircon particle which was previously determined.¹² A shift in pH_{iep} to a lower pH is indicative of adsorption of polyacrylate anion on the silicate surface.¹² A decrease in pH suspension below pH 3 redispersed the coated particles as d_{50} decreased to 2 μm .

3.2. Viscosity of 48 vol.% suspensions

3.2.1. Effect of polyacrylate concentration

Fig. 2 shows the apparent viscosity at 500 s⁻¹ as a function of the amount of polyacrylate added for different powder mixtures.

For the mixture of stoichiometric composition, the addition of approximately 0.24 wt.% of dispersant resulted in a well dispersed suspension as viscosity values decreased to a minimum (15 mPa s). Simultaneous dispersion of both components was probably due to an electrostatic stabilization mechanism as zeta potential values for the powders with polyacrylate adsorbed¹² at pH 9 increased to -30 and -100 mV for alumina and zircon, respectively.

High viscosity values were obtained at lower polyacrylate concentrations. This may be explained by an insufficient adsorption of polyacrylate anion mainly on the alumina surface due to low available dispersant content. Then, a decreased in the net negative charge of the mixed powders produced a significant increase in viscosity.

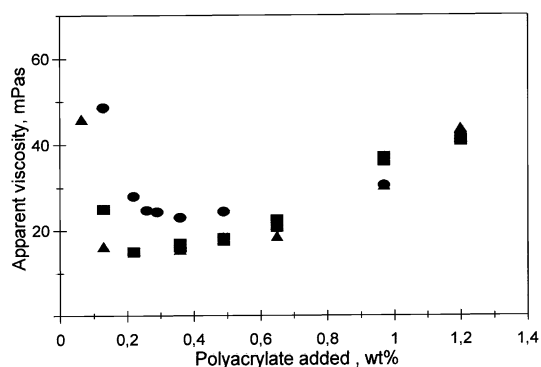


Fig. 2. Apparent viscosity at 500 s⁻¹ of 48 vol.% suspensions as a function of the amount of polyacrylate added for the different solid compositions; ■ stoichiometric, ● with more alumina and ▲ with a zircon excess powder mixtures.

Further additions than 0.65 wt.% increased gradually the apparent viscosity to 40 mPas probably due to the presence of an excess of polyelectrolyte.

Adsorption isotherm of this mixture is shown in Fig. 3. The adsorbed amount was near to $0.05\text{--}0.06\text{ mg m}^{-2}$ for dispersant contents up to 0.65 wt.%. The adsorption was low and the amount of polyelectrolyte in solution gradually increased from 2 to 7.8 g l^{-1} or four times. At added concentrations of 1–1.2 wt.%, the adsorbed amount increased to 0.124 mg m^{-2} and the equilibrium concentration up to 14 g l^{-1} . This increase in adsorption may be explained by a zeta potential decrease, due to excessive amounts of dispersant, that reduced electrostatic repulsion between the polyelectrolyte anion in solution and the negative surface sites.

Stability and rheological properties of the high solid loading suspensions that contained polyelectrolytes were very sensitive to the adsorption coverage.^{6–8} Moreover, according to these works as polyelectrolytes at alkaline pH show a low affinity adsorption behavior, large additions that are necessary to obtain the adsorption saturation limit result in high amount of free polyelectrolyte in solution.

Adsorption measurements for zircon–alumina mixtures (Fig. 3) also showed that the uptake of polyelectrolyte was low as compared with the amount remaining in solution. Above 1 wt.%, the equilibrium concentration increased up to 7 times. At alkaline pH, weak polyelectrolytes as polyacrylates are completely ionized (highly charged) and the presence of small amount in solution could increase the ionic strength of the suspension. Weak destabilization due to an excess of polyelectrolyte was previously attributed to low electrostatic repulsion resulting from a double layer compression (high ionic strength) and to a depletion flocculation mechanism.^{6–8} As attraction became significant, the formation of flocs is possible.

The variation of the viscosity with the added amount of polyacrylate may be explained as above for the other mixtures.

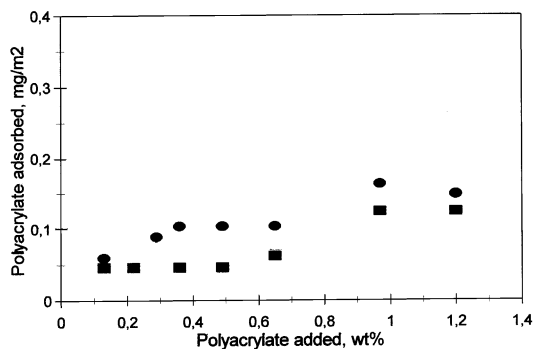


Fig. 3. Adsorption isotherm of polyacrylate on the mixed powder at pH 9.1–9.2; ■ stoichiometric, ● with more alumina.

3.2.2. Effect of the chemical composition of the mixture

Fig. 2 shows that the apparent viscosity at 500 s^{-1} of 48 vol.% suspensions as a function of the amount of polyacrylate added for different solid compositions indicates that the amount of polyacrylate required to reach the minimum viscosity decreased from 0.24 to less than 0.2 with increasing the content of zircon. The optimum amount of dispersant corresponded to that one calculated from the concentration of polyacrylate necessary to stabilize a 48 vol.% alumina suspension at pH 9 and the amount of alumina in the mixture suggesting a very low adsorption of polyacrylate on the zircon surface. As zircon particles became strongly negative at high pH, weak adsorption may be attributed to the presence of few positive surface sites and to strong electrostatic repulsion between the dominant negatively charged surface sites and polyacrylate anion in solution.

At low polyelectrolyte concentration, the suspension of the mixture which contained the highest amount of alumina showed higher viscosity values than the ones of the other compositions. On the contrary, a low viscosity suspension was obtained with the higher amount of zircon in the mixture.

At low dispersant concentration, the increase in viscosity was high for the suspension containing more alumina. For this powder composition (with higher specific surface

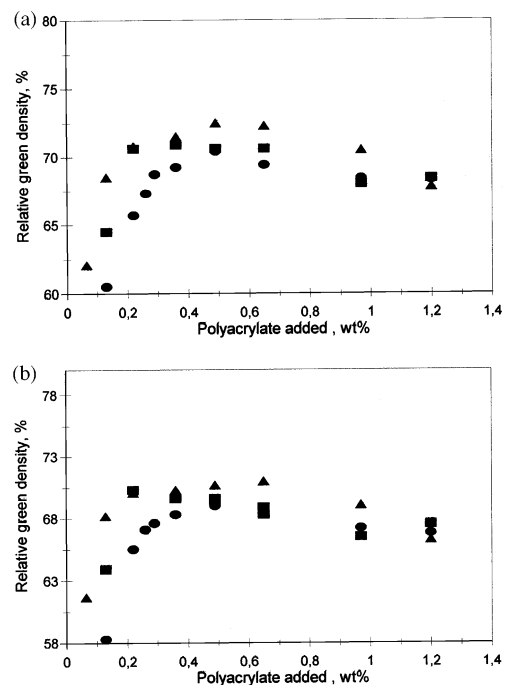


Fig. 4. Relative density of green compacts obtained from 48 vol.% suspensions by slip casting (a) and pressure filtration (b) as a function of the amount of polyacrylate added for the different solid compositions; ■ stoichiometric, ● with more alumina and ▲ with a zircon excess powder mixtures.

area) the amount of added polyacrylate probably was insufficient for effective deflocculation due to the greater polyacrylate adsorption on the alumina surface than that on the zircon powder. Therefore, at low dispersant additions high viscosity values of the suspension may be explained by an incomplete coverage of alumina surface, that reduced the net negative surface charge of the mixture in combination possibly with a heterocoagulation mechanism (electrostatic attraction between two or more particles with opposite charge).

In addition, the net negative surface charge of the mixture and electrostatic repulsion decreased with an increase in the content of alumina in the mixture, because of the lower zeta potential value of the alumina powder at pH near 9.2 than the one of the zircon. Some electrostatic attraction between the fine and neutral alumina particles and the coarse and negatively charged zircon particles (heterocoagulation) occurred. Even for similar charged particles, the barrier for flocculation between coarse and fines is lower than that for coarse-coarse particles.¹¹

For polyacrylate concentrations higher than 0.65 wt.%, the viscosity of all the alumina/zircon ratio mixtures increased to similar values. Adsorption isotherms for the mixed powder containing more alumina is also shown in Fig. 3. Although a similar behaviour was observed, the adsorbed amount of polyacrylate was slightly higher than that adsorbed for the stoichiometric compositions. The adsorbed amount was nearly constant ($0.09\text{--}0.1\text{ mg m}^{-2}$) for dispersant concentrations of 0.28–0.65 wt.%. Then, further increase in the added amount of dispersant produced an increase in adsorption to 0.15 mg m^{-2} . Nevertheless, at 1.2 wt.% the equilibrium concentration increased seven times (13 g l^{-1}) and consequently the ionic strength effect on the viscosity became significant. Although the adsorbed amount increased, free polyelectrolyte in solution reduced the colloidal stability of the suspension due to lower electrostatic repulsion and to depletion flocculation.^{6–8}

The mixed powders had different grain size distributions but the viscosity of suspensions at high dispersant additions had nearly identical values for the three compositions studied. This may be related to a poor dispersion and, consequently, to the presence of flocs that contributed to a less efficient packing of the particles.

3.3. Green density vs viscosity

Figure 4 shows the green density of compacts were obtained by slip casting in a plaster mould and by pressure filtration at 8 MPa.

Dried compacts prepared by slip casting from suspensions in the low viscosity range achieved a highest relative density of 70.5, 69.5 and 72% TD for stoichiometric, with more alumina and with a zircon excess compositions. At polyacrylate contents of 0.24–0.65

wt.% the adsorbed amount was sufficient to enhance electrostatic repulsion between particles that assisted to obtain a more efficient packing of the particles.

Bodies prepared from suspensions having lower dispersant content showed a decrease in density with respect to those obtained from a stable suspension. This result was due to low electrostatic repulsion and heterocoagulation between particles, resulting from insufficient adsorption of polyacrylate. This effect was more pronounced on the density of compacts (up to 8–10%) prepared from a suspension containing a higher amount of fine alumina particles.

Although, viscosity of the suspensions containing 0.29–0.49 wt.% of dispersant was slightly higher than the minimum, green density of the compacts was similar to the values obtained from the minimum viscosity. A possible explanation is related with a shear thinning flow behavior of these suspensions because all suspensions having a minimum viscosity showed a Newtonian behavior. A small apparent yield value can be useful in preventing some mass segregation during consolidation.

For the three compositions, density of compacts prepared from suspensions containing 0.65–1.2 wt.%, i.e. an excess of dispersant in solution, showed a relatively lower decrease (2–3%) than that produced from suspensions containing an insufficient amount of dispersant, which exhibited a similar increase in viscosity. Adsorption and zeta potential measurements indicated that dispersion of zircon alumina mixtures at pH 9 is mainly produced by an electrostatic mechanism. However, it is possible that large amounts of free polyacrylate in solution enhanced an additional steric contribution due to higher adsorbed amounts and a change in configuration of the adsorbed molecule. High ionic strength reduced ionization of weak polyelectrolytes as polyacrylate and enhanced the development of loops.⁷ The additional steric repulsion may prevent close contact between particles as interparticle distances decreased during consolidation. Thus zircon-alumina suspensions with excessive amounts of polyacrylate in solution showed a similar packing behavior to weakly flocculated suspensions, thereby producing green bodies with a relatively high density compare to those from a stable suspension. In weakly flocculated suspensions the presence of short-range repulsive potentials between particles reduce interfacial friction, facilitating particle sliding and arrangement.

Pressure filtered bodies were slightly less dense than the compacts prepared by slip casting and the density decreased with an increase in the viscosity of the suspensions.

Pressure filtered compacts prepared from low viscosity suspensions gave high green density. Green density values of 69.6, 68.7 and 70.5% TD were obtained respectively for stoichiometric, with more alumina and with a zircon excess compositions.

3.4. Sintered density and microstructure of the compacts

Colloidal processing of concentrated suspensions with submicronic particles of α -alumina and zircon produced a dense dried consolidate that can be sintered to a high density body. In this case, the development of relative high sintered density was mainly controlled by: (a) the extent of mullitization reaction, b) the relative content of zirconia ($d_{t\text{-ZrO}_2} = 6.1 \text{ g/cm}^3$ and $d_{m\text{-ZrO}_2} = 5.85 \text{ g/cm}^3$) and (d) porosity.¹³

Green probes were sintered up to 1600°C, using an increase in temperature rate of 5°C/min, and a soaking time of 2 h. In Fig. 5, the density of the sintered probes as a function of the green density were plotted for the three mixtures studied. The higher density obtained were of 3.71, 3.6 and 3.82 g/cm³ for the stoichiometric, with more alumina and with zircon excess probes respectively.

It is a clear relation between the green and the sintered density for the all compositions, showing that the high green density gave the highest sintered density.

The reaction sintering process (RS) finish in a mullite–zirconia composite with a density value is dependant on two principal factors like: (a) composition and particle size and (b) temperature schedule. Claussen,² optimized the heating schedule employing a soaking step at 1450°C, in this way using a stoichiometric mixture a density of near 3.84 g/cm³ was obtained. However, this value has also a little variation depending on the relative content of tetragonal and monoclinic zirconia. Under our sintering conditions, the stoichiometric compositions reach 97% of this theoretical density. However, as will be seen, a little content of unreacted zircon is present increasing the density of the body. This effect on density is mainly observed for the excess zircon composition, which leads to material with the higher density.

Probes with excess of alumina showed the lower density of the three mixtures.

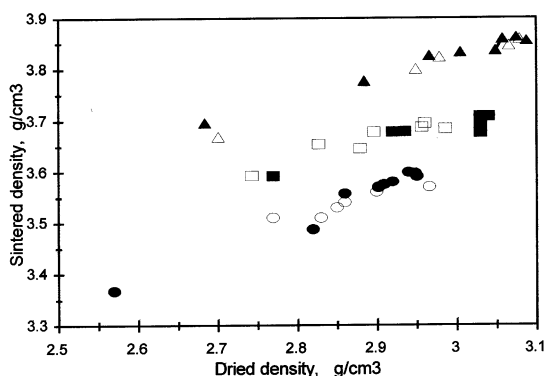


Fig. 5. Sintered density vs green density of compacts obtained from 48 vol.% suspensions by slip casting (closed symbols) and pressure filtration (open symbols) for the different solid compositions; ■ stoichiometric, ● with more alumina and ▲ with a zircon excess powder mixtures.

The density plots of Fig. 5 correspond either to slip casting or pressure filtered casts, showing that no significant differences in density between the two forming methods appeared. Although in a few percentages, sintered bodies prepared by slip casting had a higher density than that obtained using pressure filtration.

In Fig. 6, the XRD patterns of the sintered compacts are shown. For the stoichiometric compacts, mullite and zirconia (*t*-tetragonal and *m*-monoclinic) appeared as the principal phases, accompanied with unreacted zircon as a minor phase, indicating that the reaction is not completed. The presence of unreacted zircon may be explained by the heating schedule used or by its high average particle size (2 μm) if compared with that of the α -alumina (0.5 μm).

Unreacted zircon is easily observed in the excess zircon compacts. The mullite content seems to increase when the content of alumina was increased in the compositions. The ratio of tetragonal to monoclinic zirconia could not be determined.

The microstructure were analyzed on polished sections of the probes after a thermal etching at 1500°C for 10 min, as is suggested by Boch et al.³

The macrostructure texture of the sintered probes were observed with a low magnification. These appeared to be free of large defects. There was no lamination, specially in the border faces, that were in contact with the plaster or the filter. The SEM examination confirmed that there was no great heterogeneity present along the sintered materials, either in large or wide. This fact is important if it is taken in account that these mixes were only dispersed ultrasonically, to reach a good degree of dispersion, without an extra attrition milling step.

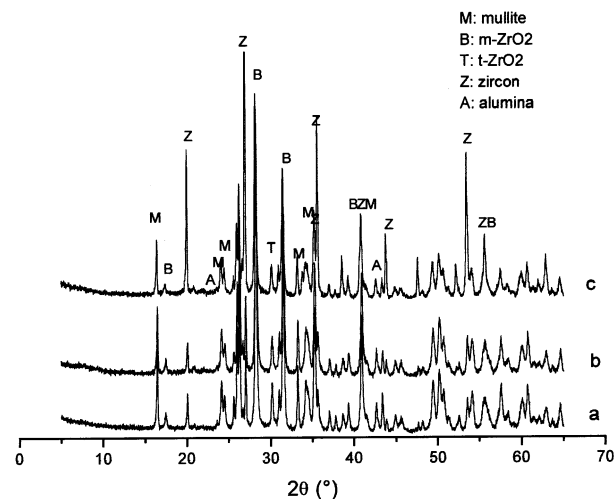


Fig. 6. XRD patterns of the sintered compacts at 1600°C for 2 h for the three compositions studied: a, with more alumina; b, stoichiometric and c, with a zircon excess powder mixtures.

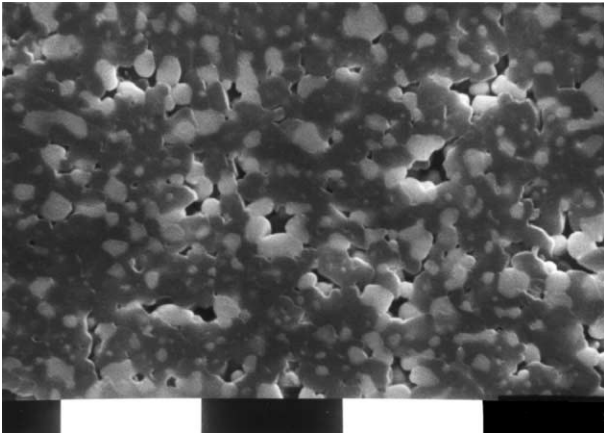


Fig. 7. Microstructure of the stoichiometric compositions after sintering at 1600°C for 2 h (scale bar = 10 μm).

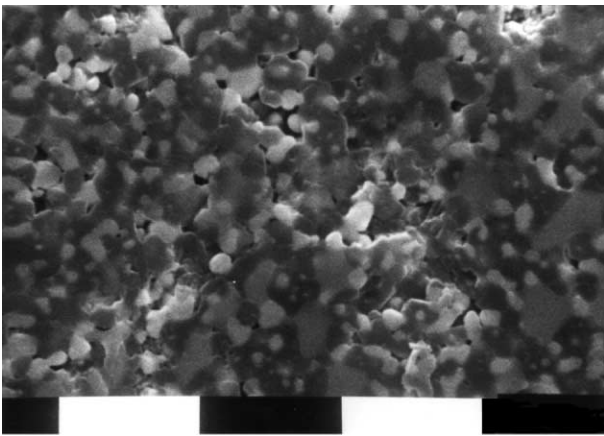


Fig. 8. Microstructure of the composition with excess of zircon, after sintering at 1600°C (scale bar = 10 μm).

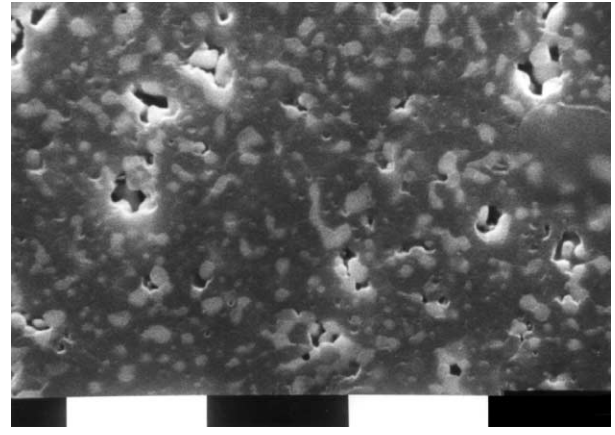


Fig. 9. Microstructure of the composition with excess of alumina, after sintering at 1600°C for 2 h (scale bar = 10 μm).

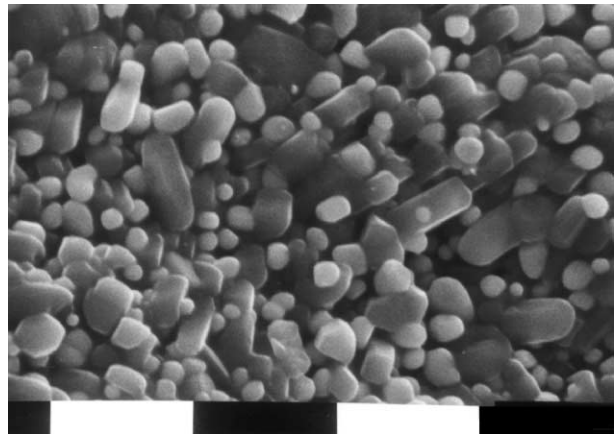


Fig. 10. Microstructure of the fracture surface of stoichiometric composition (scale bar = 10 μm).

Figs. 7–10 showed the photographs of the microstructure of the sintered bodies. The fairly dense nature of the microstructure is evident for the three materials.

The stoichiometric probe (Fig. 7) showed a microstructure that is characterized by the cross-linked structure of mullite (dark grains) and white zirconia grains. There are two types of zirconia: intergranular which were subrounded to rounded in shape and located between mullite grains and with a large size near to 2 μm; the intragranular zirconia existed within the mullite grains, are rounded in shape and smaller (> 1 μm). The large size of intergranular zirconia compared to that of intragranular grains is due to a different formation mechanism.¹³ The growth of intergranular zirconia occurred by grain boundary mass transport of zirconium ions, whereas grain growth of intragranular zirconia took place by diffusion of zirconium ions through the mullite lattice. The difference in rate of zirconium mobility was responsible for the difference in grain size.¹³

Previous studies using special XRD techniques, revealed that intragranular particles are *t*-ZrO₂, while

much of the intergranular ZrO₂ had a monoclinic crystal structure.¹⁴

The pores are well distributed and are of 3 μm or less in size.

The microstructure of the zircon excess probe is showed in Fig. 8. In this material, grey areas are in a major proportion than in the stoichiometric one, indicating that zircon (grey) is present. The pores and the phase distribution is similar to that observed in Fig. 7.

The microstructure of probe with alumina excess (Fig. 9) is very similar to the stoichiometric one, but the zirconia grains are not well defined. This material seems to have the most homogeneous microstructure than the other ones.

In Fig. 10 shows the fracture surface of the stoichiometric sintered probe in which the well formed elongated grains of mullite and the rounded grains of the intergranular monoclinic zirconia are observed. The microstructure obtained were similar to that found in composites prepared by uniaxial or isostatic compaction pressure with high thermomechanical properties.¹⁵

4. Conclusions

The degree of dispersion of different of alumina-zircon mixtures in a concentrated aqueous suspension was study using sedimentation, adsorption and rheological measurements.

Simultaneous dispersion of the powders at pH 9.1–9.2 by addition of 0.20–0.24 wt.% of polyacrylate resulted in a well dispersed suspension showing a minimum in viscosity. Adsorption of polyacrylate, mainly on alumina surface, enhanced electrostatic repulsion between both components at pH near 9. Weakly polyacrylate adsorption on zircon particles was also confirmed by d50-pH curves on diluted suspensions. A low polyacrylate concentration of 0.05 wt.% was effective to reverse positive charges on the zircon surface at pH 5 and to shift iep pH to a low value indicating an anion form adsorption.

At dispersant concentrations from 0.24 to 0.49 wt.% where the adsorbed amount was nearly constant and moderate the equilibrium concentration of free polyacrylate, high green density of the cast samples were obtained.

A relatively lower decrease in green density of compacts obtained from suspensions containing more than 0.65 wt.% may be related to an additional steric repulsion that prevent close contact of the particles as interparticle distances decreased during consolidation. These suspensions showed a similar behavior than weakly flocculated suspensions.

Optimization of the degree of dispersion of the components in the suspension improved green density of the cast samples and consequently the final density of the product. Pressure filtered bodies were less dense (~1%) than the compacts prepared by slip casting and the relative density increased slightly with decreasing the viscosity of the suspensions. After sintering at 1600°C for 2 h the samples produced bodies with a final density of 3.71, 3.6 and 3.82 g/cm³ for stoichiometric, with more alumina and with a zircon excess compositions.

Microstructures are similar to those obtained by other compaction-sintering methods which exhibited high termomechanical properties.

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