## AQUEOUS COLLOIDAL PROCESSING OF ZIRCONIA-TOUGHENED-ALUMINA CERAMICS

#### Heidy L. CALAMBÁS PULGARIN, Liliana B. GARRIDO and María P. ALBANO Centro de Tecnología de Recursos Minerales y Cerámica (CETMIC), C.C . 49 (B1897ZCA) M. B. Gonnet, Provincia de Buenos Aires, ARGENTINA. Fax 54 (0221) 471-0075.E-mail:palbano@cetmic.unlp.edu.ar

# ABSTRACT

Two  $Al_2O_3$ - $ZrO_2$  mixture preparation routes: classical powder mixing and addition of a Zr (IV) precursor solution to a well dispersed  $Al_2O_3$  suspension, were used to produce alumina  $(Al_2O_3)$ -zirconia  $(ZrO_2)$  slip cast composites. For the conventional powder mixing route, two commercial 3 mol% yttria-partially stabilized zirconia powders, 0.3 wt%  $Al_2O_3$ -doped (Al-doped Y-PSZ) and without  $Al_2O_3$  (Y-PSZ), were employed. The influence of the processing conditions on the density and microstructure development of sintered samples were investigated. The viscosity and yield stress values of  $Al_2O_3$ -ZN suspensions were markedly higher than those of  $Al_2O_3$ - Al-doped Y-PSZ and  $Al_2O_3$ -Y-PSZ ones, for all the compositions and solid loading studied and resulted in a less dense packing of cast samples. However, for the composite with 10.5 vol% ZN a high sintered density and a smaller  $ZrO_2$  grain size distribution compared with the conventional powder mixing route could be obtained.

Keywords:  $AI_2O_3 - ZrO_2$  suspensions, rheological properties, slip casting, sintering behavior.

# INTRODUCTION

Recently, zirconia-toughened-alumina ceramics have received considerable attention due to their attractive properties, including high-temperature mechanical strength, good thermal shock resistance, wear and oxidation resistance, low thermal conductivity, and the close match between their thermal expansion coefficients and those of metals (1). These properties make zirconia-alumina ceramics suitable for a variety of high demanding applications including dental screws, cutting blades, electrosurgical insulators, valve seals, body armor, pump components, oxygen sensors, dies, and prosthesis components such as hip joints (2). The zirconia grains embedded in an alumina matrix enhance the flexural strength, fracture toughness, and fatigue resistance. However, studies on the application of these materials in humid environment at low temperature have shown that the tetragonal→monoclinic transformation can also be induced at the surface of  $ZrO_2$  grains, leading to the so-called hydrothermal transformation (2) which produces a slow degradation of the composite mechanical properties. In order to reduce the  $ZrO_2$  susceptibility to the hydrothermal instability, 3 mol% yttria- partially stabilized zirconia powder doped with 0.3 wt% Al<sub>2</sub>O<sub>3</sub> can be used.

Colloidal shaping methods enable to achieve high microstructural homogeneity in green and sintered parts (1). The first step in the colloidal process, is the preparation of stable concentrated aqueous suspensions of the ceramic powders. Anionic polyelectrolytes such as ammonium polyacrylate (NH<sub>4</sub>PA) are commonly used as dispersant of ceramic powders in aqueous media.

Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> mixtures can be prepared by two routes: classical powder mixing and addition of a Zr (IV) precursor to a well dispersed Al<sub>2</sub>O<sub>3</sub> suspension. In this work, these two mixture preparation routes were used to produce concentrated aqueous Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> suspensions for slip casting. For the conventional powder mixing route, two commercial 3 mol% yttria-partially stabilized zirconia powders, 0.3 wt% Al<sub>2</sub>O<sub>3</sub>doped (Al-doped Y-PSZ) and without Al<sub>2</sub>O<sub>3</sub> (Y-PSZ) were employed. The different textural characteristics and interfacial charge properties of the two sub micron zirconia powders were believed to strongly affect the dispersion properties of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> suspensions. By using the Zr (IV) precursor route, it should be possible to obtain nano-sized ZrO<sub>2</sub> particles homogeneously distributed on the Al<sub>2</sub>O<sub>3</sub> particle surfaces. The small size of the zirconia grains would retain a large amount of tetragonal zirconia in the alumina matrix after sintering with the ability to transform under applied stress; thus, contributing to the transformation toughening mechanism operating in these composites.

In this study, a series of  $Al_2O_3$ - $ZrO_2$  formulations were prepared either by conventional powder mixing or a colloidal method. The influence of the zirconia content and the solid loading on the rheological properties of concentrated aqueous  $Al_2O_3$ - $ZrO_2$  slips were investigated. The density of green samples of the different  $Al_2O_3$ - $ZrO_2$  composites were compared and related to the degree of slip dispersion.

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In addition, the influence of the processing conditions on the density and microstructure development of sintered samples were investigated.

# EXPERIMENTAL PROCEDURE

## Raw materials and powder processing

Alumina ( $d_{50}$ =0.40 µm), 3 mol% yttria- partially stabilized zirconia with 0.3 wt% Al<sub>2</sub>O<sub>3</sub> ( $d_{50}$ =0.23 µm) and without Al<sub>2</sub>O<sub>3</sub> ( $d_{50}$ =0.64 µm) powders were used in this study. For the colloidal method, zirconium (IV)- propoxide solution (70 wt% in 1-propanol) was added dropwise to a stable alumina (A16) suspension in absolute ethanol. The compositions used to prepare Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> composites are summarized in Table 1. The numbers 10.5, 22 and 50 in the sample codes indicate the volume percent of zirconia in the composite.

Sample	Al <sub>2</sub> O <sub>3</sub>	Al-doped Y-	Y-PSZ	ZN
	(vol%)	PSZ (vol%)	(vol%)	(vol%)
10.5Al-doped Y-PSZ	89.5	10.5		
10.5Y-PSZ	89.5		10.5	
10.5 ZN	89.5			10.5
22Al-doped Y-PSZ	78	22		
22Y-PSZ	78		78	
22ZN	78			22
50Al-doped Y-PSZ	50	50		
50Y-PSZ	50		50	

Table1. Compositions used for the preparation of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> composites.

35 vol% aqueous  $AI_2O_3 - ZrO_2$  suspensions with the different compositions (Table 1) and various amounts of deflocculant (a commercial ammonium polyacrylate solution) were prepared by suspending particles; the pH was manually adjusted to be maintained at 9 with ammonia (25 %). In addition, slips with the optimum NH<sub>4</sub>PA concentration and different solid contents were prepared at pH 9 to study the influence of the solid loading on the rheological properties. Slips were cast in plaster molds into disks of diameter 2.61 cm. The consolidated disks were dried slowly in air for 24 h at room temperature and 24 h at 100  $^{\circ}$ C. The green samples were sintered

at a heating rate of 5 °C/min up to 1600 °C with a 2 h holding time at this temperature.

## Characterization techniques

The specific surface area (S<sub>g</sub>) and the particle size distribution of the powders were measured. To determinate the isoelectric point (IEP) of the powders, zeta potential measurements were taken. Zeta potential against pH curves were determined for 0.05 vol% slips of different powders: Al<sub>2</sub>O<sub>3</sub>, Y-PSZ, Al-doped Y-PSZ, 10.5 ZN and 22 ZN in the pH range of 3-10. In addition, the curves of the above mentioned powders with the optimum amount of NH<sub>4</sub>PA were determined. Steady state flow curves of the different Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> slips were performed by measuring the steady shear stress value as a function of shear rate in the range of shear rates between 0.5 to 542 s<sup>-1</sup> using a concentric cylinder at 25 °C. The density of the green compacts was determined by the Archimedes method using mercury displacement. The sintered samples were polished with a series of diamond pastes down to 1 µm. The alumina and zirconia grain sizes were measured using SEM micrographs of polished and thermally etched surfaces.

#### **RESULTS AND DISCUSSION**

#### Powder characterization

Figure 1a shows the particle size distribution curves of the  $AI_2O_3$ , Y-PSZ and Aldoped Y-PSZ powders. In Figure 1b the particle size distribution curves of the 10.5 and 22ZN powders after milling were compared with that of  $AI_2O_3$ . Alumina showed an unimodal distribution with particle diameters > 0.1 and < 0.75 µm, the more frequent particle diameters were in the range of 0.15-0.3 µm. A slightly narrow particle size distribution was found for AI-doped Y-PSZ; thus a greater volume of finer particles (diameters between 0.10 and 0.20 µm) and a lesser volume of particles with diameters in the range of 0.20-0.55 µm were observed, the more frequent particle diameter was 0.15 µm. A bimodal distribution curve was found for Y-PSZ, the more frequent particle diameters (0.37 and 0.65 µm) were greater than those of  $AI_2O_3$  and AI-doped Y-PSZ powders.



Figure 1: Particle size distribution curves of different powders: (a)  $AI_2O_3$ , Y-PSZ and AI-doped Y-PSZ; (b)  $AI_2O_3$ , 10.5 and 22ZN powders after milling.

A slightly wider particle size distribution was found for the ZN powders with respect to that of  $Al_2O_3$  (Fig. 1b); a lesser volume of finer particles (< 0.35 µm) and a greater volume of particles with diameters in the range of 0.35-0.9 µm were found. The more frequent particle diameters of 10.5ZN were in the same range as those of  $Al_2O_3$ ; whereas, for the 22ZN powder the more frequent particle diameters became greater (0.40-0.60 µm). Although the Zr precursor route avoided the formation of ZrO<sub>2</sub> aggregates on the  $Al_2O_3$  particles, it let to aggregates of some  $Al_2O_3$  particles with very fine ZrO<sub>2</sub> uniformly distributed. The diameters of the  $Al_2O_3$  aggregates increased with increasing the nano-ZrO<sub>2</sub> content. The specific surface area (S<sub>g</sub>) of  $Al_2O_3$ , Y-PSZ, Al-doped Y-PSZ, 10.5ZN and 22ZN powders was 8.74, 7.84, 12.25, 9.7 and 13 m<sup>2</sup>/g, respectively.

Figure 2 shows the zeta potential versus pH curves of the different powders. The IEP of AI-doped Y-PSZ,  $AI_2O_3$ , Y-PSZ, 10.5 and 22ZN powders was found to be 8.9, 8.5, 7, 6 and 4.3, respectively. The  $AI_2O_3$  and Y-PSZ IEPS were in agreement with those reported in the literature for these powders (1); however, the IEP of AI-doped Y-PSZ powder was close to that of  $AI_2O_3$  and higher than that of Y-PSZ. Thus, the  $AI_2O_3$  surface coating shifted the  $pH_{IEP}$  of Y-PSZ from 7 to 8.9. On the contrary, the very fine zirconia particles homogeneously distributed on the  $AI_2O_3$  particles surface shifted the  $pH_{IEP}$  of AI- $O_3$  from 8.5 to 6 for 10.5ZN and from 8.5 to 4.3 for 22ZN. The  $pH_{IEP}$  decreased with increasing the ZN content and for 22ZN the  $pH_{IEP}$  became similar to that reported for ZrO<sub>2</sub> which was about 4-6 (3).



Figure 2: Zeta potential vs. pH curves of different powders:  $AI_2O_3$ , Y-PSZ, Al-doped Y-PSZ, 10.5 and 22 ZN.

## Optimum NH<sub>4</sub>PA concentration and zeta potential for the different compositions.

The optimum NH<sub>4</sub>PA concentration, i.e. that which gave slips with the lowest viscosity, was determined at pH 9 for the slips with the different compositions (Table 2). The optimum NH<sub>4</sub>PA concentration decreased from 0.34 to 0.25, 0.4 to 0.21 and 0.52 to 0.11 wt% by substituting 10.5, 22 and 50 vol% of Al-doped Y-PSZ by Y-PSZ in the mixtures, respectively. The minimum viscosity occurred at 0.3 and 0.4 wt% NH<sub>4</sub>PA solution for the 10.5 and 22ZN powders, respectively. For the mixtures with the same composition, the viscosity values were lower for the slips prepared with Y-PSZ; whereas, the ZN slips exhibited the highest viscosity values

Table 2. Optimum NH<sub>4</sub>PA concentration for the different compositions of  $AI_2O_3$ -ZrO<sub>2</sub> suspensions at pH 9

Sample	Optimum NH₄PA	Slurry viscosity
	concentration (wt%)	(mPa.s at γ=542 s <sup>-1</sup> )
10.5Al-doped Y-PSZ	0.34	8.1
10.5Y-PSZ	0.25	6.6
10.5 ZN	0.30	11.0
22Al-doped Y-PSZ	0.40	9.0
22Y-PSZ	0.21	6.7
22ZN	0.40	11.7
50Al-doped Y-PSZ	0.52	12.5
50Y-PSZ	0.11	4.0

Figure 3a shows the zeta potential versus pH curves of different powders:  $AI_2O_3$ , 22AI-doped Y-PSZ and 22Y-PSZ dispersed in slips with the optimum amount of NH<sub>4</sub>PA solution added. In Figure 3b the zeta potential versus pH curves of the ZN powders in suspensions with the optimum amount of NH<sub>4</sub>PA solution added are compared with that of  $AI_2O_3$ .



Figure 3: Zeta potential versus pH curves of different powders dispersed in slips with the optimum amount of  $NH_4PA$  solution added: (a)  $AI_2O_3$ , 22AI-doped Y-PSZ and 22Y-PSZ; (b)  $AI_2O_3$ , 10.5ZN and 22ZN.

The adsorption of NH<sub>4</sub>PA shifted the pH<sub>IEP</sub> of Al<sub>2</sub>O<sub>3</sub> (Fig. 2) from pH 8.5 to 3.1; the IEP of the mixtures 22Y-PSZ and 22Al-doped Y-PSZ was found to be 4.25 and lower than 3, respectively. The strong adsorption of the polyelectrolyte on the mixture 22Al-doped Y-PSZ resulted in an increase in the magnitude of the negative zeta potential in the pH range 3.25-10.1. The higher pH<sub>IEP</sub> of the mixture 22Y-PSZ with respect to that of 22Al-doped Y-PSZ and Al<sub>2</sub>O<sub>3</sub> was a consequence of the lower adsorption of the polyelectrolyte. The zeta potential value at pH 9 was -45.5, -57.3 and -62.7 mV for Al<sub>2</sub>O<sub>3</sub>, 22Y-PSZ and 22Al-doped Y-PSZ, respectively (Figure 2a). The IEP of 10.5 and 22ZN was found to be 3.4 and 2.2, respectively. Similar negative zeta potential values of the 22ZN slips with and without NH<sub>4</sub>PA at alkaline pH were observed as a consequence of the lower adsorption of the polyelectrolyte (Figs. 2 and 3b).

#### Rheological properties

Figure 4a shows the viscosity at 542 s<sup>-1</sup>versus the zirconia content for stabilized slips at pH 9 with different solid loading. The measured flow curves were satisfactorily fitted with the Casson model (R=0.99). The Casson model equation is:

$$\tau^{1/2} = \tau_0^{1/2} + (\eta_p \gamma)^{1/2}$$
 (A)

where  $\tau$  is the shear stress,  $\gamma$  is the shear rate,  $\tau_0$  is the yield stress and  $\eta_p$  represents the limiting viscosity at a high shear rate range. The particles in a flocculated suspension form floc groups or a network, because of the mutual attraction between particles; the aggregated particles make the suspension difficult to flow, thereby increasing the  $\tau_0$  value (4). Consequently, the  $\tau_0$  value of the Casson model could be used as a parameter that indicated the degree of aggregation and consequently the degree of slip flocculation. The effect of zirconia content on the  $\tau_0$  value for different solid loading is shown in Figure 4b. For Al<sub>2</sub>O<sub>3</sub> – ZN and Al<sub>2</sub>O<sub>3</sub> - Al-doped Y-PSZ slips, the viscosity and  $\tau_0$  values increased with increasing the zirconia content in the mixtures; while for Al<sub>2</sub>O<sub>3</sub> - Y-PSZ slips, a decrease in the viscosity and  $\tau_0$  with increasing Y-PSZ content were found. For all the concentrations, the  $\tau_0$  values of Al<sub>2</sub>O<sub>3</sub> – ZN slips were markedly higher than those of Al<sub>2</sub>O<sub>3</sub> - Al-doped Y-PSZ and Al<sub>2</sub>O<sub>3</sub> - Y-PSZ ones; these differences became larger as the zirconia content increased. The  $\tau_0$  values of 48 vol% slips prepared from 22ZN, 22 Al-doped Y-PSZ and 22Y-PSZ decreased from 6.5 to 2.2 and 1.1 Pa, respectively.



Figure 4: Viscosity at 542 s<sup>-1</sup> (a) and  $\tau_0$  (b) versus the zirconia content for stabilized Al<sub>2</sub>O<sub>3</sub>-Y-PSZ, Al<sub>2</sub>O<sub>3</sub> – Al-doped Y-PSZ and Al<sub>2</sub>O<sub>3</sub> –ZN slips at pH 9 with different solid loading.

The important resistance to flow for the  $Al_2O_3 - ZN$  slips was attributed to the presence of some aggregated particles. As the degree of aggregation increased with increasing ZN content, the difference in the  $\tau_0$  values between the  $Al_2O_3 - ZN$  slips and those prepared from the different zirconia powders tended to be more pronounced with increasing the zirconia content. Besides, an important increase in the slopes of the  $\tau_0$  curves with increasing solid loading were found for the different slips due to the higher frequency of the collisions between separate particles, increasing the resistance to flow. Therefore, the difference in the  $\tau_0$  values between the different and the solid loading. The degree of aggregation of the 10.5 and 22ZN particles had more effect on the  $\tau_0$  than on the viscosity values "as expected". Since the negative zeta potential of the ZN slips were high, the high viscosity values of these slips could be attributed to the presence of some aggregated particles which increased the quantity of entrapped liquid not available for flow.

A significant increase in the viscosity values of  $Al_2O_3$  - Al-doped Y-PSZ slips with respect to  $Al_2O_3$  -Y-PSZ ones was found for all the compositions and solid loading studied (Fig. 4a). As was previously shown (Fig. 3a) the negative zeta potential at pH 9 for the slips with the same content of Y-PSZ and Al-doped Y-PSZ were similar and high enough to impart stability to the suspensions. The higher viscosity values of the suspensions prepared from Al-doped Y-PSZ could be explained taking into account the particle effective size concept (5). The calculated volume fraction of solids corresponded to the solid loading of the slurries and did not consider hydrated and adsorbed layers around the particles. Since the specific surface area of the Al-doped Y-PSZ particles was markedly higher than that of  $Al_2O_3$  and Y-PSZ, a greater hydration surrounding the Al-doped Y-PSZ particles was expected. Besides, the substitution of  $Al_2O_3$  and/or Y-PSZ by Al-doped Y-PSZ in the mixtures increased the effective solid volume fraction at a given solid loading and decreased the amount of free-liquid available for flow, thereby increasing the viscosity values.

#### Characterization of green and sintered samples

Figure 5 shows the green density of cast samples prepared from 48 vol%  $AI_2O_3$  -Y-PSZ,  $AI_2O_3$  - AI-doped Y-PSZ and  $AI_2O_3$  – ZN slips versus zirconia content. As the

slip viscosity decreased a more dense packing of the samples could be obtained (Figs. 4a and 5).



Figure 5: Green density of cast samples prepared from 48 vol%  $AI_2O_3$  -Y-PSZ,  $AI_2O_3$  - Al-doped Y-PSZ and  $AI_2O_3$  – ZN slips versus the zirconia content in the mixtures.

For Al<sub>2</sub>O<sub>3</sub> - Y-PSZ slips, green density values between 64.3 % (10.5Y-PSZ) and 65.3 % (50Y-PSZ) of theoretical density were found; while for Al<sub>2</sub>O<sub>3</sub> - Al-doped Y-PSZ slips, the green density had values between 63.2 % (10.5Al-doped Y-PSZ) and 60.0 % (50Al-doped Y-PSZ) of theoretical density. Thus, the substitution of Y-PSZ by Al-doped Y-PSZ in the Al<sub>2</sub>O<sub>3</sub> – ZrO<sub>2</sub> mixtures increased the slip viscosity with NH<sub>4</sub>PA resulting in a less dense packing of cast samples. The cast samples obtained from the Al<sub>2</sub>O<sub>3</sub> – ZN slips exhibited the lowest green density values: 57.0 % and 54.0 % of the theoretical density for 10.5 and 22ZN, respectively.

The sintered density of the composites with 10.5 vol%  $ZrO_2$  was compared. Similar sintered density values of 97.3, 97.8 and 97.7 % of the theoretical density were found for 10.5ZN, 10.5Al-dopedY-PSZ and 10.5Y-PSZ, respectively. The fine  $ZrO_2$  particles homogeneously distributed on the Al<sub>2</sub>O<sub>3</sub> particle surfaces increased the densification rate and a high sintered density could be achieved. Figure 6 shows the zirconia grain size distribution curves obtained for the different composites. The 10.5Al-dopedY-PSZ curve was slightly shifted to lower grain diameters with respect to the 10.5Y-PSZ curve; the more frequent grain diameters were 0.6 and 0.7 µm for 10.5Al-dopedY-PSZ and 10.5Y-PSZ, respectively. The Zr (IV) precursor route let to a narrower and smaller zirconia grain size distribution compared with the conventional powder mixing route. The ZrO<sub>2</sub> grain diameters were in the range 0.1-0.8 µm and the more frequent grain diameter was 0.4 µm.

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Figure 6: Zirconia grain size distribution curves obtained for the different composites with10.5 vol% ZrO<sub>2</sub> sintered at 1600 °C.

#### CONCLUSIONS

Two Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> mixture preparation routes: classical powder mixing and addition of a Zr (IV) precursor solution to a well dispersed  $Al_2O_3$  suspension, were used to produce Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> slip cast composites. For the conventional powder mixing route, two commercial 3 mol% yttria-partially stabilized zirconia powders, 0.3 wt% Al<sub>2</sub>O<sub>3</sub>-doped (Al-doped Y-PSZ) and without Al<sub>2</sub>O<sub>3</sub> (Y-PSZ), were employed. The rheological properties of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> suspensions prepared with the different zirconia powders and the Zr (IV) precursor, and their particle packing behaviour during slip casting were compared. The viscosity and yield stress values of Al<sub>2</sub>O<sub>3</sub> - ZN slips were markedly higher than those of Al<sub>2</sub>O<sub>3</sub> - Al-doped Y-PSZ and Al<sub>2</sub>O<sub>3</sub> - Y-PSZ ones; these differences became larger with increasing both the zirconia content and the solid loading. The high resistance to flow for the  $AI_2O_3 - ZN$  slips was attributed to the presence of some aggregated particles; the degree of aggregation increased with increasing ZN content from 10.5 to 22 vol%. The cast samples obtained from the Al<sub>2</sub>O<sub>3</sub> – ZN slips exhibited the lowest green density values; however, for 10.5 ZN a high sintered density and a smaller zirconia grain size distribution could be achieved. The small ZrO<sub>2</sub> grains would lead to a high portion of tetragonal phase retained in the alumina matrix after sintering with the ability to transform under applied stress.

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