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Direct coagulation casting of YSZ powder suspensions using MgO as coagulating agent

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Abstract

Direct coagulation casting (DCC) of aqueous 8 wt% yttria stabilized zirconia (YSZ) powder suspensions prepared using ammonium poly(acrylate) dispersant has been studied using MgO as coagulating agent. Small amount (<0.1 wt% based on YSZ) of MgO powder dispersed in the YSZ powder suspension at ~5 °C set the suspension in to stiff wet-coagulated body when exposed to room temperature (30 °C) due to the reaction between ammonium poly(acrylate) and MgO. MgO concentration equivalent to react with dispersant did not coagulate the YSZ powder suspension though it precipitate the whole ammonium poly(acrylate) dispersant as Mg-poly(acrylate). This is because of the ability of the YSZ powder to disperse in water at alkaline pH (~9.5) without any dispersant by electrostatic mechanism. The YSZ powder suspensions form stiff coagulated bodies at MgO concentration double or more of the equivalent amount required for reacting with the dispersant. Setting of the YSZ powder suspension is due to the heterocoagulation of the YSZ particles and MgO particles added in excess of the equivalent amount to react with the dispersant, having opposite surface charges. The wet-coagulated body showed relatively high compressive yield strength (155 kPa) and Young's modulus (3.1 MPa). The green bodies prepared by humidity controlled drying of the wet-coagulated bodies sintered to >98% TD at 1550 °C.

Keywords: A. Suspension; A. Shaping; D. ZrO_2; D. MgO; Direct coagulation casting

1. Introduction

Direct coagulation casting (DCC) is a well-recognized method for near-net-shape fabrication of ceramic components from concentrated aqueous ceramic powder suspensions [1]. In DCC the powder suspensions cast in a mould is set in to stiff coagulated bodies by destabilizing the suspensions by producing acid, base or electrolyte *in situ* from water soluble precursor molecules present in the suspension medium [1–20]. Generation of ammonia (base) and electrolyte like ammonium bicarbonate and ammonium carbonate from urea by urease catalyzed *in situ* hydrolysis is well studied for setting of most of the ceramic powder systems such as alumina, zirconia, silicon carbide silicon nitride, etc. [1–12]. The ammonia coagulates the powder suspensions, prepared in acidic medium, by pH shift near the iso-electric point. On the other hand, ammonium bicarbonate and ammonium carbonate coagulate the powder suspensions by compressing the electrical double layer. *In situ* generation of acid from acid anhydrides, esters, lactones and hydroxyl aluminium acetate has also been reported for destabilization of powder suspensions, prepared in alkaline medium, by pH shift for DCC [13–20].

Recently, we have reported a new DCC process for aqueous alumina suspensions using MgO as coagulating agent. In this, the Mg^{2+} ions produced from sparingly soluble MgO react with the un-adsorbed ammonium poly(acrylate) dispersant present in the suspension medium and form precipitate of Mg-poly(acrylate) [21–23]. This shifts the dispersant adsorption equilibrium towards the desorption side. This leads to insufficient dispersant coverage on alumina particle surface and resulted in coagulation of the suspension. In addition, the precipitated Mg-poly(acrylate) acts as binder and provides strength and stability to the coagulated body. The alumina suspensions coagulate at MgO concentrations equivalent to react with the dispersant [21–23].

The present work reports DCC of aqueous yttria stabilized zirconia (YSZ) powder suspensions prepared using ammonium poly(acrylate) dispersant. Novelty of this work is that the

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setting of the YSZ powder suspensions in to stiff coagulated bodies is not due to desorption of the ammonium poly(acrylate) dispersant from the YSZ particle surface alone, but due to heterocoagulation of YSZ particles and MgO particles used in excess of the equivalent amount to react with the dispersant, having opposite surface charges.

2. Experimental

8 wt% YSZ powder of average particle size 0.4 μm and surface area 2.8 m^2/g was procured from Aldrich Chemicals, USA. A 35 wt% aqueous solution of ammonium poly(acrylate) was used as dispersant. Analytical reagent grade light MgO powder (Thomas Baker, Mumbai) of surface area 32.1 m^2/g was used as coagulating agent. The MgO powder contains agglomerates of flaky particles of size nearly 2–3 μm [22]. Ammonia solution and nitric acid used for pH adjustments were procured from Merck India Ltd. A silicon free defoaming agent (BYK-012, BYK Chemie, GmbH, Germany) was used to avoid foaming of the suspension during milling. Distilled water was used for preparation of the powder suspensions.

Aqueous YSZ powder suspensions (45 vol%) were prepared by tumbling the zirconia powder, water and various concentrations of the dispersant in polyethylene containers along with zirconia grinding media. The pH of the suspensions was adjusted to ~9.4 using ammonia solution. After 12 h of tumbling the viscosity of the suspensions were measured using a Brookfield viscometer with a small sample adapter and cylindrical spindle (SC-21) to determine the optimum amount of dispersant required for dispersion of the powder.

The YSZ powder suspensions (45 vol%) prepared at optimum dispersant concentration were cooled in an ice bath to a temperature of ~5 °C. Various amount of MgO powder was added to the ice cold zirconia suspensions and thoroughly mixed by ball milling at the ice cold condition for 20 min. In order to maintain low temperature (~5 °C) during milling, the slurries present in smaller polythene containers were packed in bigger polyethylene containers with crushed ice and then subjected to milling on a roller mill. Immediately after mixing, viscosity of the slurries was measured at various times. The viscosity measurement was taken using SC-28 spindle at a shear rate of 2.8 s^{-1} and the slurries were exposed to room temperature (~30 °C) during the viscosity measurements.

The YSZ powder suspensions of 48 vol% solids loading were used for DCC studies. 0.1 wt% (based on YSZ powder) defoaming agent was added to the powder suspensions used for casting to prevent foaming of the suspension. The suspensions mixed with required amount of MgO at low temperature was cast in closed stainless steel moulds to prepare cylindrical coagulated bodies of 22 mm diameter and 45 mm length. The coagulated bodies removed from the mould were subjected to compressive stress–strain (Hounsfield, S-sires, UK) measurement in a universal testing machine at a cross-head speed of 2 mm/min. The compressive strength (yield stress) and Young's modulus of the wet-coagulated bodies were obtained from the stress–strain graph [8,6].

The coagulated YSZ bodies removed from the mould were dried at room temperature at ~75% relative humidity. The humidity condition was created in a desiccator using saturated sodium chloride solution. The coagulated bodies dried at room temperature to a constant weight were then heated in an air oven at 120 °C to remove the residual moisture. Shrinkage of the coagulated body during drying was calculated from its dimensions before and after drying. Density of the dried coagulated body was calculated from the weight and dimensions. The green bodies were sintered at 1550 °C for 2 h. The heating rate used was 5 °C/min. Density of the sintered YSZ ceramic was determined by Archimedes method. Microstructure of the green and sintered bodies was observed on fractured and polished surfaces, respectively using a scanning electron microscope (LEO 1455, UK).

3. Results and discussion

It is well known that ammonium poly(acrylate) forms well dispersed aqueous ceramic powder dispersions at pH in the range of 9–9.5 [24,25]. Therefore, in the present work, YSZ powder suspensions were prepared at a pH of nearly 9.4. Optimization of dispersant concentration for forming a well-dispersed powder suspension is the primary step in any colloidal processing route. Fig. 1 shows effect of concentration of dispersant on viscosity of 45 vol% aqueous YSZ powder suspensions. The aqueous YSZ powder suspension without the ammonium poly(acrylate) dispersant at ~9.4 pH shows low viscosity of ~0.425 Pa s. This indicates that YSZ powder disperses at this pH by electrostatic mechanism. However, the viscosity of the suspension decreases with the addition of ammonium poly(acrylate) dispersant and reaches a minimum at dispersant concentration of nearly 0.075 wt%. Unlike alumina suspension, addition of dispersant above the optimum level drastically increased the viscosity of the suspension [21]. However, dispersant concentration higher than the optimum amount is desirable for DCC using MgO because the MgO powder needs to be initially dispersed along with the YSZ by the dispersant [21–23]. Therefore, dispersant concentration of 0.1 wt% is used for preparation of YSZ powder suspensions for

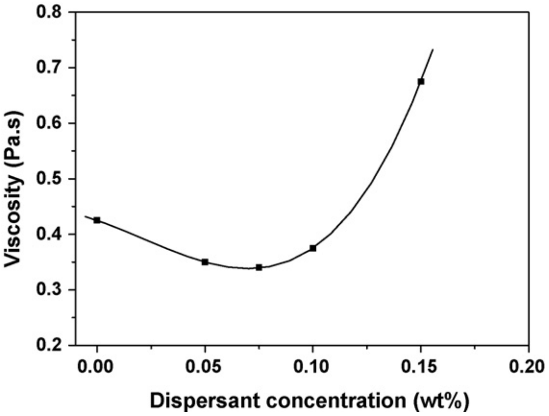

Fig. 1. Effect of concentration of dispersant on viscosity of 45 vol% aqueous YSZ powder suspensions.

all further studies though the viscosity is slightly higher than that of the dispersion at optimum dispersant concentration.

It has been observed in our earlier studies with alumina suspensions that the MgO concentration equivalent to react with the dispersant set the suspensions by coagulation [21,22]. The amount of MgO equivalent to react with the dispersant was calculated by considering the fact that two monomer units in ammonium poly(acrylate) react with one MgO. However, in the case of YSZ powder suspension, addition of MgO equivalent to react with the ammonium poly(acrylate) dispersant (~0.023 wt%) at room temperature did not coagulate the powder suspension even after 24 h. Though the YSZ powder suspension did not coagulate at 0.023 wt% MgO, the pH increased from ~9.4 to 9.8 within 30 min due to the ammonium hydroxide generated by the reaction between MgO and ammonium poly(acrylate) [21]. On the other hand, addition of 0.046 wt% MgO converts the YSZ powder suspension in to a stiff coagulated paste within 20 min. The rapid coagulation poses difficulties in proper mixing of the suspension with MgO and subsequent casting in moulds. It has been noticed in our earlier studies that mixing of alumina powder suspensions with MgO at low temperature of ~5 °C keeps the viscosity of the suspensions un-changed for long time due to decrease in the rate of reactions leading to coagulation due to lower solubility of MgO at low temperature [22]. Therefore, in the present work, MgO powder is mixed with the YSZ powder suspension at low temperature of ~5 °C.

Fig. 2 shows viscosity variation with time of the YSZ powder suspension mixed with various MgO concentrations at low temperature (~5 °C) when exposed to room temperature (30 °C). At 0.023 wt% MgO (equivalent to react with the dispersant) the viscosity of the suspension remains un-changed even after 24 h. However, at 0.046 wt% MgO, the viscosity of the suspension showed a marginal increase from 0.5 to 1 Pa s in 10 min. After 10 min, the suspension viscosity rapidly increased with time and reached a value of 47 Pa s at the end of 18 min. The viscosity further increased with time and the slurry becomes a stiff paste within 30 min. However, the viscosity measurement could not be possible with the present viscometer. At 0.069 wt% MgO, the suspension viscosity increased from 0.75 to 4.5 Pa s in 9 min and then to 48 Pa s in 11 min. In a DCC process the suspension viscosity should remain un-changed for sufficient time to carryout the degassing and casting operations. It has been confirmed that the viscosity of the YSZ suspensions after mixing with the MgO could be maintained more or less constant over a period of 30 min by keeping the slurry in an ice bath. Therefore, degassing before casting could be done by applying vacuum by keeping the slurry in an ice bath.

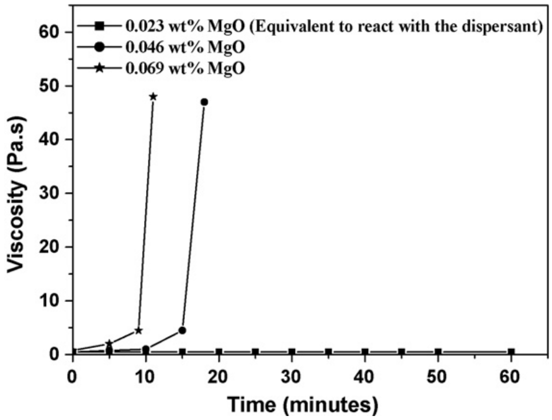

Fig. 2. Viscosity variation with time of the YSZ powder suspension mixed with various MgO concentrations.

When MgO is mixed with the YSZ powder suspension at low temperature (~5 °C) the ammonium poly(acrylate) present in the dispersion medium adsorbs on the MgO particle surface and disperse them along with the YSZ particles. When the slurry is exposed to room temperature the solubility of MgO increases and produces Mg^{2+} ions at higher rate. These Mg^{2+} ions react with the ammonium poly(acrylate) to form Mg-poly(acrylate). This leads to the desorption of dispersant from the particle surface. At MgO concentration of 0.023 wt% (equivalent to react with dispersant), though the whole ammonium poly(acrylate) is desorbed from the particle surface, the suspension did not undergoes coagulation. This is due to the fact that the YSZ powder disperses in water at alkaline pH (~9.5) without any dispersant due to the electrostatic mechanism. However, at MgO concentrations of 0.046 and 0.069 wt%, the dispersant reacts with only 0.023 wt% MgO and leaves 0.023 and 0.046 wt%, respectively of MgO in the suspension. It has been reported that iso-electric point of aqueous MgO powder suspension is nearly 10.8 [26]. That is, the MgO particles show relatively high positive charge below 10.8 pH [26]. It has also been reported that the iso-electric point of YSZ is less than 8 [27]. That is, the YSZ particles show negative surface charge at pH above 8. It appears from the viscosity increase with time that the reaction between the ammonium poly(acrylate) dispersant and MgO is tending to complete within 10 min after exposure of the slurry to room temperature. This exposes the bare surface of remaining unreacted MgO particles. The rapid viscosity increase after 10 min is due to heterocoagulation of YSZ and MgO particles having opposite surface charges at this suspension pH (~9.8).

In order to confirm the heterocoagulation process, various amounts of MgO is added to 20 vol% YSZ powder suspension at pH ~ 9.4 containing 0.1 wt% dispersant and measured the particle size of the YSZ in the suspension after 1 h of mixing. At MgO concentration of 0.023 wt% (equivalent to react with the dispersant) the median particle size (0.407 μm) and particle size distribution observed is almost same as that of the original powder indicating that no heterocoagulation takes place. However, at 0.046 and 0.069 wt% MgO the particle size distribution graph shifted to higher particle size range. The median particle size of YSZ in the suspension at MgO concentration of 0.046 and 0.069 wt% are 2.18 and 3.75 μm, respectively. Fig. 3 shows particle size distribution of the YSZ powder in the suspension containing various concentrations of MgO.

The DCC slip should have low viscosity and yield stress before casting to facilitate easy mould filling. Fig. 4 shows viscosity at various shear rate of 48 vol% YSZ powder

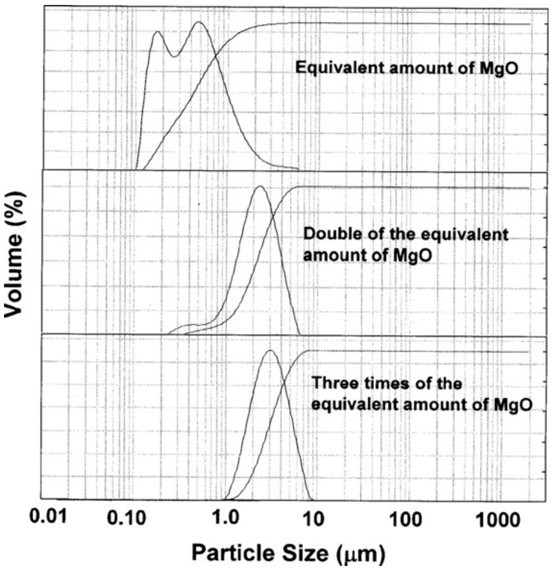

Fig. 3. Particle size distribution of the YSZ powder in the suspension containing various concentrations of MgO.

suspension containing 0.046 and 0.069 wt% MgO for DCC. The YSZ DCC suspensions showed pseudoplastic flow behaviour. The YSZ powder suspension containing 0.069 wt% MgO showed higher viscosity and yield stress than that containing 0.046 wt% MgO. The YSZ DCC suspensions containing 0.046 and 0.069 wt% MgO showed viscosity in the range of 1–0.21 and 2–0.38 Pa s, respectively at shear rate in the range of 4.65–93 s^{-1}. The corresponding yield stress values calculated using Casson model are nearly 3.6 and 7.4 Pa, respectively [28]. The higher viscosity and yield stress of DCC slip prepared at 0.069 wt% MgO is attributed to premature coagulation due to higher rate of reaction between MgO and the ammonium poly(acrylate) dispersant at higher concentration of MgO. Therefore, further casting studies were conducted at MgO concentration of 0.046 wt%.

The YSZ DCC slip containing 0.046 wt% MgO cast in cylindrical mould could be removed without any deformation in 1 h after casting. Fig. 5 shows stress–strain plot of the wet-coagulated YSZ body prepared at 0.046 wt% MgO. The wet-coagulated body undergoes deformation mainly elastically up to the yield point. However, plastic deformation is observed after the yield point. The average compressive strength and Young's modulus of the wet-coagulated body is ~155 kPa and 3.1 MPa, respectively. During the stress–strain measurement, cracks are observed on wet-coagulated body near the yield point indicating its brittle nature. Fig. 6 shows photograph of wet-coagulated YSZ bodies after and before the compressive stress–stain measurement.

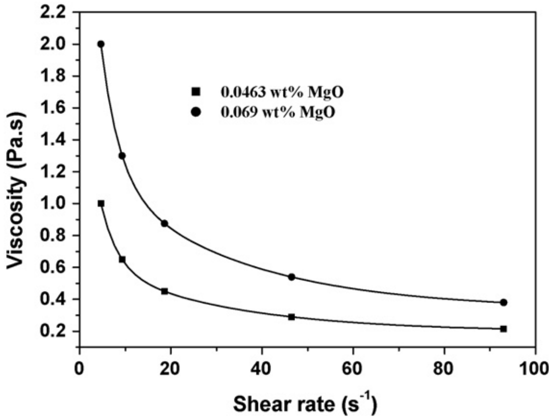

Fig. 4. Viscosity at various shear rates of 48 vol% YSZ DCC slips.

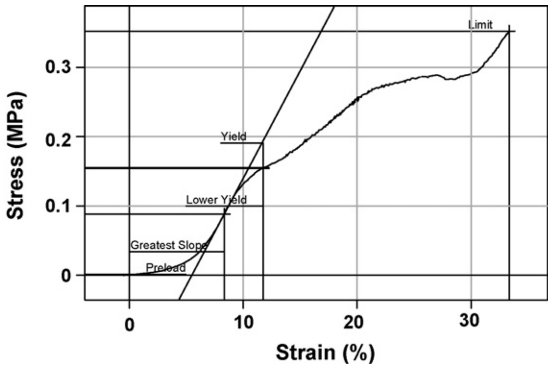

Fig. 5. Stress–strain plot of the wet-coagulated YSZ body prepared from the DCC slip containing 0.046 wt% MgO.

In the case of alumina, the strength and stability of wet-coagulated bodies increased with increase in ammonium poly(acrylate) concentration due to binding of the alumina particles by the nanoparticle Mg-poly(acrylate) formed by the reaction between ammonium poly(acrylate) and MgO [22]. The wet-coagulated alumina bodies achieve sufficient strength and stability against the oscillatory stresses at ammonium poly (acrylate) concentration above 0.8 wt% [22]. On the other hand, in the YSZ powder suspensions, incorporation of ammonium poly(acrylate) above 0.1 wt% is difficult due to drastic increase in viscosity of the suspension with dispersant concentration after the optimum dispersant concentration. The amount of Mg-poly(acrylate) present is too low to explain the relatively high strength (~155 kPa) of wet-coagulated YSZ body formed by the DCC process. The high strength of wet-coagulated YSZ

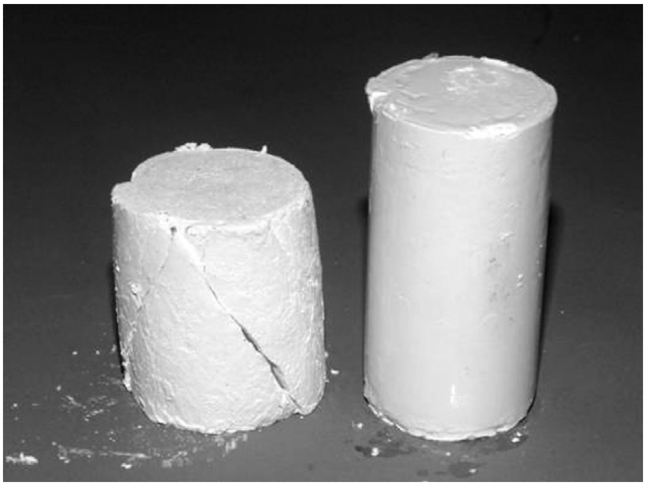

Fig. 6. Photograph of wet-coagulated YSZ bodies after and before the compressive stress–strain measurement.

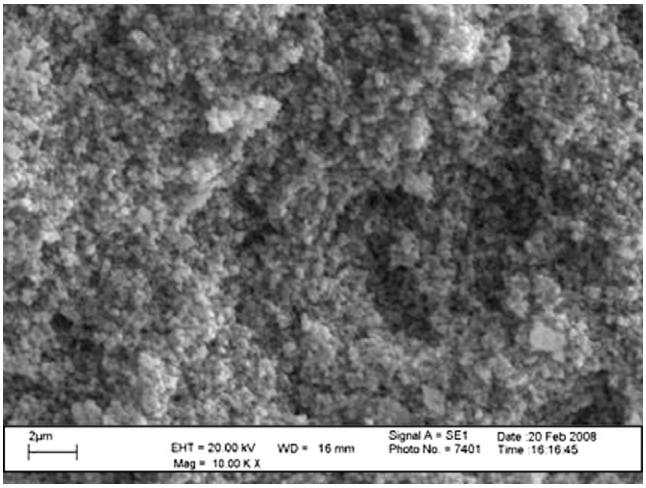

Fig. 7. SEM photomicrograph of the fractured surface of the YSZ green body prepared by the DCC.

body could be due to strong cohesive force between YSZ particle and MgO having opposite surface charges. It has been reported that the strength of wet-coagulated bodies increases with decrease in homogeneity of particle network [29]. Heterocoagulation of YSZ and MgO particles would results in a coagulated body with less homogeneous particle network that contributes to its high strength.

Room temperature (~30 °C) drying of the cylindrical wet-coagulated YSZ bodies at 75% relative humidity did not produce any crack or deformation. The coagulated body showed a linear shrinkage of 2.54% during drying. The green body obtained by drying the wet-coagulated body showed a density of 52.4% TD. Microstructure of the fractured surface of the YSZ green body showed uniformly packed YSZ particles. Fig. 7 shows SEM photomicrograph of the fractured surface of the YSZ green body. The YSZ green body prepared by the coagulation casting process sintered to ~98% TD at 1550 °C. Linear shrinkage observed during sintering is nearly 20.2%. Fig. 8 shows SEM photomicrograph of the polished and thermally etched surface of the sintered YSZ ceramic prepared by the DCC process. Average grain size calculated from the microstructure by linear intercept method is 1.2 μm.

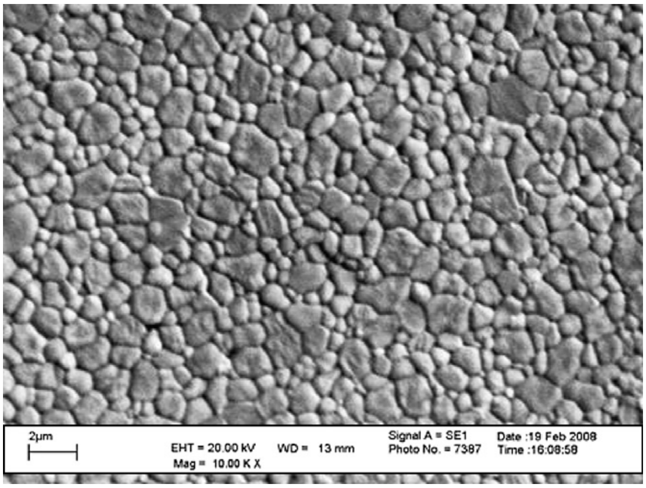

Fig. 8. SEM photomicrograph of the polished and thermally etched surface of the sintered YSZ ceramic prepared by the DCC process.

4. Conclusions

Small amount (<0.1 wt% based on YSZ) of MgO powder dispersed in the YSZ powder suspension at ~5 °C coagulated the suspension in to stiff paste when exposed to room temperature (~30 °C) due to the reaction between ammonium poly(acrylate) dispersant and MgO. MgO concentration higher than the equivalent amount required to react with dispersant is needed to set the YSZ powder suspension in to stiff coagulated body. The setting of the YSZ suspension is due to heterocoagulation of YSZ particles and MgO particles (added in excess of the amount required to react with the dispersant) having opposite surface charges at the processing pH. The wet-coagulated body showed relatively high compressive strength (155 kPa) and Young's modulus (3.1 MPa). The green bodies prepared by humidity controlled drying of the wet-coagulated bodies sintered to >98% TD at 1550 °C.

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