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Direct coagulation casting of yttria-stabilized zirconia using magnesium citrate and glycerol diacetate

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Abstract

Direct coagulation casting via controlled release of high valance counter ions (DCC-HVCI) has been reported in recent years. In this paper, concentrated yttria-stabilized zirconia (YSZ) suspensions were coagulated using DCC-HVCI method with magnesium citrate as coagulating agent assisted by pH shift in the presence of glycerol diacetate. The effect of ammonium polyacrylate (PAA-NH_4) on the dispersibility of YSZ powder was investigated. The influence of concentrations of glycerol diacetate and magnesium citrate on pH and viscosities of YSZ suspensions was studied. The results indicate that concentrated YSZ suspensions can be coagulated by adding 2 vol% glycerol diacetate and magnesium citrate above 0.5 wt% at room temperature for 2–5 h. The compressive strength of coagulated wet samples is above 2.0 MPa. YSZ ceramics sintered at 1450 °C show homogeneous microstructures with relative densities of 98.9–99.2%. Flexural strength of YSZ ceramics is 869 ± 84 MPa.
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Keywords: Direct coagulation casting; High valance counter ions; YSZ; Magnesium citrate; Glycerol diacetate

1. Introduction

Yttria-stabilized zirconia (YSZ) ceramics have been widely used as an advanced structural material for its high strength and toughness and good wear resistance [1–4]. It is desirable to obtain a homogeneous microstructure and to fabricate components with complex shapes for various applications. Colloidal processing is recognized as an ideal route to prepare near-net-shape ceramics with tailoring rheological properties, minimal organic binder content, high wet strength, and excellent microstructural homogeneity [5,6]. New colloidal processing methods such as gel casting, direct coagulation casting (DCC) have been applied to form green bodies from well-dispersed zirconia suspension [7–11]. Direct coagulation casting is a novel method for near-net-shape preparation of ceramic parts from concentrated aqueous ceramic powder suspensions by introducing the DLVO theory into ceramic colloidal forming. The concentrated suspension is destabilized by the decomposition producing acid, base or electrolyte in situ from water soluble precursor molecules present in the suspension to either shift the pH of the suspension to the isoelectric point (IEP) of the ceramic powder system or increase the ionic strength of the suspension [12–15]. However, there are some drawbacks such low wet strength (~100 kPa), long coagulation time (1–3 days) and easy cracking which have hindered DCC as a promising colloidal forming method in industrial application [16,17].

According to the Schulze–Hardy rule, the destabilizing power of an electrolyte is due mainly to the valence of its counter ion. The destabilizing power of counter ion can be determined by measuring the critical coagulation concentration (CCC) of an electrolyte which is the minimum concentration required to rapidly coagulate a given suspension. The DLVO theory can be used to derive an expression which relates the CCC to the counter ion valence. CCC=constant/z^6, where z is the charge on the counter ion. For z=1,2 and 3 the ratio of the CCCs are $1/1^6$ (1), $1/2^6$ (0.016) and $1/3^6$ (0.0014), respectively [18–22]. Based on this concept, we proposed a novel ceramic forming method called direct coagulation casting via controlled release of high valence counter ions (DCC-HVCI) with a combination of the DLVO theory and the Schulze–Hardy rule. High valence counter ions (Ca^{2+}, Mg^{2+}, SO_4^{2-}, etc.) were

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controlled release via increasing temperature which can destabilize high solid loading alumina suspension [23–27]. Complex ceramic components have been manufactured with high sintered density and uniform microstructure via the above methods. However, the release of high valance counter ions is controlled by heating the suspensions. Heating is unavoidable during the coagulation process which can be a problem for preparing large and thick-cross section ceramic parts owing to uneven heating. Unequal heating may cause inhomogeneous coagulation which impacts the properties of ceramics. It was reported that magnesium citrate shows solubility sensitive to pH in the alkaline region. Magnesium citrate has minor dissolution in aqueous solution at room temperature at pH above 10, but the solubility increases gradually with the decrease of pH [28]. Previous reports also showed that hydrolysis of glycerol diacetate shifted the pH of suspension from 10 to 9 at room temperature [27]. The combination of the two compounds has been applied to coagulate concentrated alumina suspension at room temperature [29].

The aim of this paper is to study direct coagulation casting of YSZ suspension via controlled release of high valence counter ions (DCC-HVCI) using magnesium citrate as coagulating agent and glycerol diacetate as pH controller at room temperature. The effect of dispersant on the dispersibility of YSZ suspensions was investigated by measuring the viscosity and the zeta potential of the suspensions. Influence of concentrations of glycerol diacetate and magnesium citrate on the pH and rheological properties of YSZ suspensions was evaluated. YSZ green bodies and sintered ceramics have been prepared by DCC-HVCI and the properties of the samples were measured.

2. Experimental procedure

2.1. Materials

Commercially available tetragonal zirconia powders stabilized with 3 mol% Y_2O_3 (OZ-3Y, Guangdong Orient Zirconic Ind. Sci. & Tech. Co., Ltd., China) with $d_{50}=0.13$ μm and surface area 8.53 m^2/g was used for suspension preparation. Solution with 30 wt% ammonium polyacrylate (PAA-NH_4) purchased from Zibo Jinghe Chemical Dyestuff Co., Ltd., China was used as dispersant. Chemical purity magnesium citrate [$Mg_3(C_6H_5O_7)_2 \cdot 10H_2O$] with 99.0% purity was used as coagulating agent. Glycerol diacetate (GDA) was used to tailor the pH of the suspension. The molecule weights of magnesium citrate and glycerol diacetate are 703.4 and 176.17, respectively. Both of magnesium citrate and glycerol diacetate were purchased from Beijing Hengye Zhongyuan Chemical Co., Ltd. Deionized water was used in all preparation process.

2.2. Coagulation process

Suspensions with different solid loadings were prepared by mixing the YSZ powder, water and ammonium polyacrylate in polyethylene containers for 24 h. Zirconia balls with diameter of 5–10 mm were used as grinding media. The mass ratio between grinding media and zirconia powder is 1:2. Hydrochloric acid and ammonia were utilized to adjust the pH of the suspensions. The suspensions were degassed in vacuum condition for 15 min at room temperature. Different amounts of magnesium citrate were added to the suspensions and mixed thoroughly by continuing the tumbling process for another 20 min to obtain homogenous suspensions. Then glycerol diacetate was added into the suspensions. The prepared suspensions were cast into a plastic mold with a rubber seal. Coagulated wet samples were demolded after resting at room temperature for 2–5 h. The green body was dried at 80 °C for 24 h and then was sintered at 1450 °C for 2 h at heating rate of 5 °C/min.

2.3. Characterizations

The zeta potential of YSZ suspensions was measured by a zeta potential analyzer (CD-7020, Colloidal Dynamics, USA). The solid loading of the suspension was 10 vol%. 1 M HCl and NaOH solutions were used to adjust the pH of the suspension. The measurement was performed at room temperature. The pH value of YSZ suspension was measured by a LE438 pH meter (Mettler, USA). In order to determine the critical coagulation concentration of magnesium for 50 vol% YSZ suspension, different amounts of magnesium were added to the suspension. 1 mol/L magnesium chloride was used for the experiment. The pH of the suspension is adjusted to 9.2 before the measurement. The rheological properties of the suspension were measured using a KINEXUS rheometer (Malvern Instruments, Worcestershire, U.K.) attached with C25 R0634 SS spindle and PC25 C0138 AL cylinder with shear rates in the range of 0.1–1000 s^{-1}. To test the compressive strength wet samples, cylindrical bodies with 25.5 mm in diameter and a height between 25 and 30 mm were cast. The compressive strength of wet samples and flexural strength of sintered ceramics was measured by an AG–IC20KN (Shimadzu, Japan) testing machine with a crosshead speed of 0.5 mm/min. There were 10 samples for each measurement. Flexural strength was measured with a three-point bending technique. The sintered samples were cut and polished before strength measurement. The density of the green samples was determined on the cut and fine ground rectangular bars by measuring the dimensions and weight. The density of the sintered samples was measured using the water displacement technique. The microstructure of green bodies and sintered samples was observed by a SSX–550 (Shimadzu, Japan) scanning electron microscope (SEM).

3. Results and discussion

3.1. Preparation of high solid loading and low viscosity YSZ suspension

High solid loading and low viscosity suspension is desirable for colloidal processing to avoid defects generated from agglomerates and to decrease the possibility of cracking and deformation during sintering due to high shrinkage. As a polyelectrolyte, ammonium polyacrylate has been used to disperse aqueous ceramic powder suspension in the pH range of 9–9.5 [30–32]. In order to prepare a well-dispersed zirconia suspension, suitable amount of ammonium polyacrylate should be considered. Fig. 1 shows the effect of concentration of ammonium polyacrylate on

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