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Feature article

In-situ coagulation of yttria-stabilized zirconia suspension via dispersant hydrolysis using sodium tripolyphosphate

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ABSTRACT

A novel in-situ coagulation method without coagulation agent and adjusting pH value for yttria-stabilized zirconia (YSZ) suspension via dispersant hydrolysis is reported. Sodium tripolyphosphate (STPP) is used as dispersant to prepare electrostatic stabilized YSZ suspension. Influences of STPP contents on the dispersion and pH value of YSZ suspension were investigated. It indicated that there was a well-dispersed YSZ suspension with the addition of 0.3 wt% STPP at pH = 10. Influence of coagulation temperature on coagulation process and properties of green body was investigated. The sufficiently high viscosity suspension to coagulate was achieved at $60-80\,^{\circ}$ C. The coagulation mechanism was different from traditional direct coagulation casting. The suspension was coagulated by directly shifting the isoelectric point to the original state without increasing the ionic strength and adjusting the pH value. It was proposed that the YSZ suspension could be destabilized via decrease of zeta potential by sodium tripolyphosphate hydrolyzing at elevated temperature. Coagulated samples with wet compressive strength of 3.60 MPa could be demolded without deformation by treating 50 vol% YSZ suspension with 0.3 wt% STPP at $60\,^{\circ}$ C for 30 min. Dense YSZ ceramics with flexural strength of $887\pm110\,^{\circ}$ MPa and relative density of 98.9% had been prepared by this method sintered at $1450\,^{\circ}$ C for 3 h.

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1. Introduction

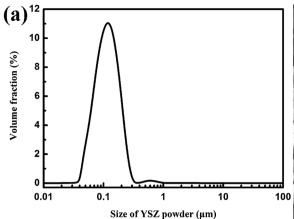
Yttria-stabilized zirconia ceramics have been widely used as an advanced structural material due to the excellent performances such as high strength, high toughness, good wear resistance, high thermal expansion coefficient and low thermal conductivity [1–4]. In various applications, YSZ ceramics components with complex shape and homogeneous microstructure are highly demanded. Direct coagulation casting (DCC) is a promising and effective method for the fabrication of complex shape ceramic parts which is based on the idea of that a high solid loading suspension is destabilized by a time-delayed internal reaction using a catalyst [5–7]. The suspension can be coagulated by either shift pH to isoelectric

http://dx.doi.org/10.1016/j.jeurceramsoc.2017.05.044 0955-2219/© 2017 Elsevier Ltd. All rights reserved. point (IEP) or increase the ionic strength into suspension [8,9]. Up to now, there are enormous amounts of investigation on DCC of YSZ ceramic [10-12].

In the previous studies, dense non-oxide ceramics including both silicon nitride and silicon carbide ceramics had been prepared by direct coagulation casting via dispersant reaction [13,14]. In this method, the non-oxide ceramic suspensions were coagulated via the decrease of zeta potential by an acid-base reaction between basic dispersant and an acid which was hydrolyzed from coagulation agent. Although there were many advantages of dispersant reaction and other colloidal forming methods, some corrupt practices of these methods for aqueous suspension were not avoided due to the addition of coagulation agents, such as using slightly soluble coagulation agent, non-uniform coagulation, long coagulation time, superfluous organic additives and so on [15–17]. Sodium tripolyphosphate (STPP) is a kind of polyphosphates which includes ammonium polyphosphate (APP) and sodium hexametaphosphate (SHMP) etc. There are wide applications of polyphosphate as water

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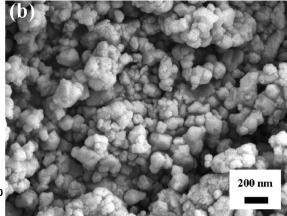


Fig. 1. (a) Particle distribution and (b) microstructure of YSZ powder.

retention agent, pH regulator, detergent builder and metal-chelator to be employed in food processing, waste water treating and other commercial productions [18]. It is also used as dispersant to prepare suspension [19–21]. A large amounts of research focused on the chemical properties of polyphosphate and the application of sodium tripolyphosphate hydrolysis in industrial production [22–24].

In the present work, the chemical properties of dispersant are still used in experiment. The YSZ suspension with high solid loading and low viscosity was prepared using sodium tripolyphosphate as dispersant. It was coagulated by dispersant hydrolysis method which abandoned the addition of coagulation agent. The influences of coagulation temperature on zeta potential, pH value and rheological properties of YSZ suspension with STPP were investigated. The YSZ suspension could be directly coagulated in-situ without any coagulation agents and pH regulators at elevated temperature. A novel in-situ coagulation method for YSZ suspension was proposed by controlling the dispersant hydrolysis with temperature increase.

2. Experimental

2.1. Materials

Fig. 1 shows the particle distribution and microstructure of YSZ powder. Tetragonal zirconia powder (OZ-3Y, stabilized with 3 mol% Y_2O_3) with average particle size of 0.13 μ m and specific surface area of 8.53 m²/g was produced from Guangdong Orient Zirconic Ind. Sci. & Tech. Co., Ltd., China. Sodium tripolyphosphate was used as dispersant with different concentrations. Na₄P₂O₇, NaH₂PO₄ and Na₂HPO₄ were used in proportion as the hydrolysates of sodium tripolyphosphate to investigate the effect of hydrolysates on viscosity of alumina suspension. All of the chemicals were analytic reagent and produced from Sinopharm Chemical Reagent Co., Ltd., China. Also, deionized water was used in the preparation of suspension.

2.2. Preparation of YSZ ceramics

Fig. 2 shows the flowchart of the in-situ coagulation process via dispersant hydrolysis. Different solid loadings YSZ suspensions were prepared by tumbling the YSZ powder, dispersant, water and grinding media for 24 h. Grinding media was zirconia balls with diameter of 5–10 mm. The mass ratio between YSZ powder and grinding media was 2:1. The suspensions were degassed for 15–20 min under vacuum condition. Then, the YSZ suspensions were cast into plastic molds. The molds were placed in a water bath

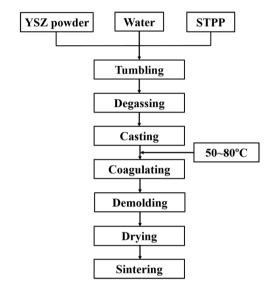


Fig. 2. Flowchart of the in-situ coagulation process via dispersant hydrolysis.

which was set a heating rate of $5\,^{\circ}$ C/min. The heating was lasted a period of time at different temperatures and then demolded. The samples were dried at $80\,^{\circ}$ C for 24 h. Finally, the green bodies were sintered at $1450\,^{\circ}$ C for 3 h.

2.3. Characterizations

LE438 pH meter (Mettler, Toledo, Switzerland) was employed to measure the pH value of YSZ suspension. Zeta Potential Analyzer (CD-7020, Colloidal Dynamics Co., Ltd., Ponte Vedra Beach, FL, USA) was employed to measure the zeta potential of YSZ suspensions with a stirring speed setting at 300 r/min. 10 vol% YSZ suspension was used in the test. HCl (1 mol/l) and NaOH (3 mol/l) aqueous solutions were prepared and used to adjust the pH value of the suspensions. In order to analyze the influence of pH value on zeta potential of alumina suspension, the ionic strength and conductivity of the suspension were maintained a constant via adding NaCl (analytic reagent). Rheometer (KINEXUS PRO, Malvern Instruments, Worcestershire, UK) attached with PC25 C0138 AL cylinder and C25 R0634 SS spindle was employed to measure the rheological properties of the YSZ suspensions. FTIR spectra were collected on Vertex 70 v Fourier Spectrophotometer (Bruker, Germany) using pressed KBr pellets as reference substance. AG-IC20KN (Shimadzu, Tokyo, Japan) testing machine with a cross head speed of 0.5 mm/min was employed in the wet com-

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