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Effect of the molecular weight and concentration of PEG solution on microstructure and mechanical properties of Si₃N₄ ceramics fabricated by gel-casting

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ABSTRACT

Gel-casting is a promising preparation technology of Si₃N₄ structural ceramics. The process involves drying of the “green” gel-cast parts before densification. And the drying of green gel-cast bodies is an important step in the gel-casting manufacturing process. In this work, the Si₃N₄ gel-cast green bodies were dried in polyethylene glycol (PEG) solution with the purpose of obtaining Si₃N₄ ceramics with good mechanical properties. The effect of the molecular weight and concentration of PEG solution on drying rate, microstructure and mechanical properties of Si₃N₄ ceramics was studied. The results indicated that with the increase of molecular weight of PEG, the drying rate increased obviously and the structure became more uniform and dense when the concentration of solution was 20 wt%. The Si₃N₄ ceramics after sintering have the excellent flexural strength (662.6 MPa) under PEG600 drying condition. Furthermore, the concentration of PEG600 solution had a positive effect on drying and sintering of the green body. Therefore, the bending strength reached 871.1 MPa under 65 wt % PEG 600 solution drying condition. Overall, the drying process (drying in 65 wt% PEG600 solution) promotes the efficiency and quality of drying of Si₃N₄ gel-cast green bodies, which is beneficial for the subsequent drying and sintering process.

1. Introduction

Gel-casting is a new near-*net*-shape ceramic forming process, which is used in manufacturing advanced structural and functional ceramic parts for various industries such as turbine components for the aerospace and other industries [1–4]. The technology combines the knowledge of polymer chemistry with traditional ceramic molding technology, which greatly improves the current ceramic forming process. The gelcasting process involves the preparation of an aqueous slurry of ceramic powder, which contains small quantities of monomers, cross-linkers, sintering aids, initiators and catalysts. Gelation is initiated after the slurry is poured into a simple or complex-shaped mold. The macromolecule network that is produced by in-situ polymerization keeps the ceramic particles together. After removal from the mold, and a homogeneous wet cast part is acquired, which contains moisture about one-fourth of its mass which is then discharged by drying. After drying, the polymer gel removal and densification are executed. In comparison with traditional ceramic forming processes, the gel-casting has the advantage of requiring little machining, faster

casting and a less-expensive densification process [4]. In the past 20 years, gel-casting has been widely used in composite materials, porous materials and functional materials and other fields [5,6]. However few practical applications have been found on industrial scale [7,8]. One of the main reasons is the uncontrollable defects which originated from the drying process of green bodies [9].

The drying of pure gels is a time consuming, complex process [10] due to the slow transport of liquid through the porous solid medium which reduces the effectiveness of manufacturing. Conventional methods in drying ceramics prepared by gel-casting, using drying air or gas with particular conditions of humidity and temperature, is usually confronted with difficulties [11]. Non-uniform and differential drying in various areas due to the solvent gradient, induces structural and residual stresses which cause defects, such as cracking, warpage, bending and the other malformations. In addition, in order to avoid defects, the drying rate cannot be too fast (especially for a thick-walled body with large size), which results in low efficiency of this drying method. Studies conducted by HARN et al. [12] have shown that the time required for complete drying of green bodies with this method is at

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least 24 h. Therefore, a new drying method should be employed in the premise of guarantying the same drying effect.

Liquid desiccant drying [13] which had previously been used for wood drying is proposed by JANNEY and KIGGANS [14]. When the green body is immersed into a liquid desiccant, the water is excluded from the body because of the small water gradient existing between the solvents and the green body. The moisture in the cross-linked polymer gels which has three-dimensional network structures migrate to the solvents to comply the purpose of drying. Compared with conventional air drying methods, liquid desiccant provides a more uniform medium which allows no region of the part to be dried too quickly resulting in the reductions of the residual stresses. And the buoyant support [9] of the gelled body during the drying is another useful feature of liquid desiccant drying to effectively reduce the appearance of the deformation and cracking, especially in the case of large bodies with different wall thicknesses. BARATI et al. [11] used an aqueous solution of polyethylene glycol (PEG1000) as the liquid desiccant for Al_2O_3 green bodies, which resulted in the removal of 30% moisture in 3 h. TRUNEC [15] investigated the influence of the molecular mass of various PEG on the drying efficiency. A. Barati [11,16] and so on used PEG solution as a liquid desiccant to dry the aluminum oxide body obtained through gel-casting, and achieved good results. At the same time, in the liquid desiccant drying, the green body loses safely about 20–30 wt% of interior solvent (water) in 2–3 h, which is considered a breakthrough in the critical stage of the drying [17]. However, the effect of liquid desiccant drying on the Si_3N_4 gel-cast body has not been studied further.

In the present work, the aim was to promote the efficiency and quality of drying of Si_3N_4 gel-cast green bodies. PEG solution was used

as the liquid desiccant for liquid drying, and the effect of the molecular weight and concentration of PEG solution on drying rate, microstructure and mechanical properties of Si_3N_4 ceramics was studied in details.

2. Experimental

2.1. Raw materials

Commercially available $\alpha\text{-Si}_3\text{N}_4$ powders (purity > 93%, $d_{50} = 0.5 \mu\text{m}$, Beijing Unisplendor Founder High Technology Ceramic Co., Ltd. China) were used as the raw materials. Al_2O_3 (Xiong Chemical Co., Ltd. China) and Y_2O_3 (Sinopharm Chemical Reagent Co., Ltd., China) powders were used as sintering aids. The raw materials used in the AM-MBAM system were summarized in Table 1.

2.2. Fabrication of the composites

Fig. 1 was the schematic diagram of gel-casting process for Si_3N_4 ceramics. Firstly, these monomer, dispersant and cross-linker were dissolved in deionized water at 45°C in the water bath to attain a premixed solution. Secondly, the premixed solution was adjusted to $\text{pH} = 9.5\text{--}10.5$ by adding $\text{NH}_3\cdot\text{H}_2\text{O}$ and HCl , then the solution was mixed with 85 wt% $\alpha\text{-Si}_3\text{N}_4$, 5 wt% Al_2O_3 and 10 wt% Y_2O_3 . Thirdly, mixing with zirconia balls (double mass of the ceramic powders) in the ball grinding mill (YXQM-4 L, China), and then the suspensions were ball-milled for 4 h (the rotating speed was 200 r/min) to promote dispersion and admixing process. In order to reduce the obstacles of the

Table 1
Raw materials for the AM-MBAM system.

Raw material	Function	Chemical formula	Manufacturer
Acrylamide (AM)	Monofunctional Monomer	$\text{C}_2\text{H}_3\text{CONH}_2$	Sinopharm Chemical Reagent Co., Ltd., China
N,N'-methylenebisacrylamide (MBAM)	Bifunctional monomer	$(\text{C}_2\text{H}_3\text{CONH}_2)_2\text{CH}_2$	Sinopharm Chemical Reagent Co., Ltd., China
Sodium hexametaphosphate	Dispersant	$\text{C}_4\text{H}_{13}\text{NO}$	Sinopharm Chemical Reagent Co., Ltd., China
Ammonium persulfate (APS)	Initiator	$(\text{NH}_4)_2\text{S}_2\text{O}_8$	Guangdong Xi-long science co., Ltd., China
N,N,N',N'-tetramethylethylenediamine (TEMED)	Accelerator (catalyst)	$\text{C}_6\text{H}_{16}\text{N}_2$	Sinopharm Chemical Reagent Co., Ltd., China
Aqua ammoniae	PH modifier	$\text{NH}_3\cdot\text{H}_2\text{O}$	Sinopharm Chemical Reagent Co., Ltd., China
Deionized water	Solvent	H_2O	

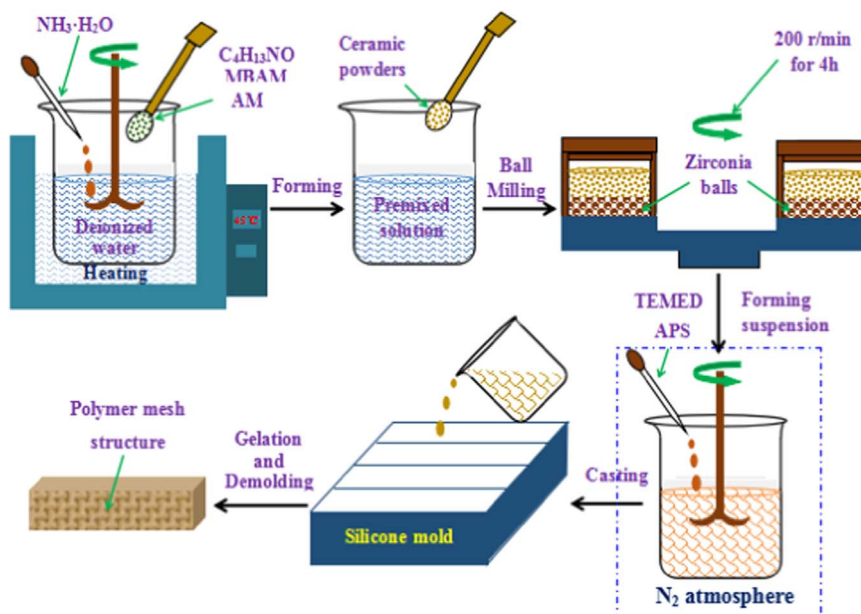


Fig. 1. Schematic diagram of gel-casting process for Si_3N_4 ceramics.

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