



Preparation of a ceramic core with high strength using an inorganic precursor and the gel-casting method



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ABSTRACT

A new process for the preparation of a ceramic core with high formability and fracture strength is proposed. It involves the use of powders coated with inorganic binders, and gel-casting. Two types of powders were used to investigate the effect of the starting material on the mechanical properties (the green and sintering strength values) of the ceramic core, according to the components and particle sizes. Both the green and sintering strengths of the core samples prepared in Case II exhibited reasonable values, due to the long gelation time and the effective glassification of the inorganic precursor, respectively; a strength of about 10 MPa was achieved after 1 h heat treatment at 1000 °C. Furthermore, the prepared core samples completely dissolved in 40 wt.% NaOH solution. Specific experimental conditions were optimized for the application of precision casting, for example, the composition ratio (of the starting powders), the inorganic precursor, and the gel-casting method.

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1. Introduction

Conventionally, a ceramic core used for hollow components in investment casting or precision casting is prepared by an injection molding method from a mixture of organic compounds (wax and surfactants) and starting powders based on zircon flour ($ZrSiO_4$) and fused silica (SiO_2) [1,2]. Polymer materials, such as paraffin wax and crystal wax, are added to form the green body of the core, and surfactants are used to maximize blending between the starting powders and the wax. However, polymer materials that have long chains of ultra-high molecular weight do not mix easily with the powders due to the high viscosity, despite the addition of surfactants. This results in a decline in the formability of the core and easily induces collapse of the green body before heat treatment. In addition, the fracture strength after heat treatment arises only from the effect of sintering between particles. This is due to the volume shrinkage and shape deformation of the core. Although much research has focused on preparing the core with good mechanical properties and with no shape deformation [3–6], these studies were limited to considering variations in the composition ratio (of the starting powders), heat treatment temperature, etc., within the investment casting process.

In the present study, the gel-casting method and an inorganic precursor were used to fabricate a ceramic core with reasonable green strength and sintering strength; here, the fracture strength of the core after heat treatment is simply referred to as the sintering strength.

The shape of the core created using the gel-casting process is fixed through the network structure of the polymer generated by the gelation of a prepolymer (a monomer and a dimer), which results in the enhancement of the green strength in the core. The inorganic precursor was used to increase the sintering strength of the core, due to the glass phase formed on the surfaces of the starting particles [7]. The prepolymer and inorganic precursor added with the liquid phase would be particularly effective in improving the mechanical properties.

2. Experimental procedure

2.1. Materials and methods

Commercially available fused silica, zircon flour, and silica (of various particle sizes) were used as starting ceramic powders to prepare the core. The composition ratios of the starting powders are given in Table 1. Tetraethyl orthosilicate (TEOS; Sigma-Aldrich Korea, Yongin, Korea) and sodium methoxide (NaOMe, Sigma-Aldrich Korea, Yongin, Korea) were used as the SiO_2 and Na_2O precursors, respectively. The following materials (all from Sigma-Aldrich Korea, Yongin, Korea) were used as additives in the gel-casting process: monomer (acrylic acid; N,N-dimethyl-3-oxo-butanamide; ethylene carboxamide), dimer (bisacrylamide), initiator (homotaurine, ammonium sulfate, ammonium peroxydisulfate), and catalyst (N,N,N'-trimethylethylenediamine and N,N,N',N'-tetramethylethylenediamine).

Formulations used to prepare the core samples under various conditions are given in Table 2. First, the starting powders were mixed

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Table 1
Composition ratios of starting powders used in Cases I and II.

Starting powder	Case I (wt.%)	Case II (wt.%)
Zircon flour 1	10	10
Zircon flour 2	10	10
Fused silica	0	10
Silica powder (2 μm [*])	10	10
Silica powder (45 μm [*])	30	25
Silica powder (149 μm [*])	30	25
Silica powder (100–200 μm [*])	10	10

* Numbers in parentheses indicate the average particle size of the added silica.

with the inorganic precursor, and then the mixture was filtered. The precursor-coated powders were dried at 80 °C for 24 h. Thereafter, the precursor-coated powders and prepolymers (monomer and dimer) were blended in a suitable solvent for the homogeneous dispersion of the two-phase heterogeneous materials. After the addition of initiator and catalyst, the mixture became a gel. The gelation time was indirectly measured by varying the temperature of inner system during polymerization. The gelled core sample was dried at 25 °C for 48 h. This formed the green body of the core. The green body was then heat-treated at 1000 °C for 1 h. Details of the fabrication process for the preparation of the core are given in Fig. 1.

2.2. Characterization

The strength of the core samples before and after heat treatment was measured using a universal testing machine (Instron 5566; Instron Corporation, Norwood, MA, USA) in the four-point mode, at a rate of 0.5 mm · min⁻¹. Five runs were performed to determine the standard deviation of the strength. The fracture morphology in the core samples after heat treatment at 1000 °C was observed using a scanning electron microscope (SEM; model JSM-5610; JEOL, Tokyo, Japan). Elemental analysis of samples was carried out using an energy dispersive X-ray spectrometer (energy resolution 133 eV) (Oxford Instruments, Oxford, UK). Dissolubility of the core samples was checked using a 40 wt.% NaOH solution at 50 °C for 10 h.

3. Results and discussion

In this study, the gelation and heat treatment processes were used to enhance the green and sintering strengths. The prepolymer and inorganic precursor with the liquid phase were used for the homogeneous mixing of the heterogeneous materials selected for use. In the polymerization of prepolymers with two or more functionalities, the polymer chains grow into a network. As the polymerization proceeds, the viscosity of this system increases and gelation commences. Furthermore, the temperature in the system increases during polymerization because chain growth is an exothermic reaction. Gelation begins immediately at the starting point of the highest temperature. Therefore, a stronger gel network is synthesized at longer gelation times. This, in turn, leads to improvement in the green strength of the core.

Experimental details pertaining to the gelation time and temperature used for the starting powders of different composition ratios are shown in Fig. 2. In Case II (Fig. 2(b)), the gelation time was longer and

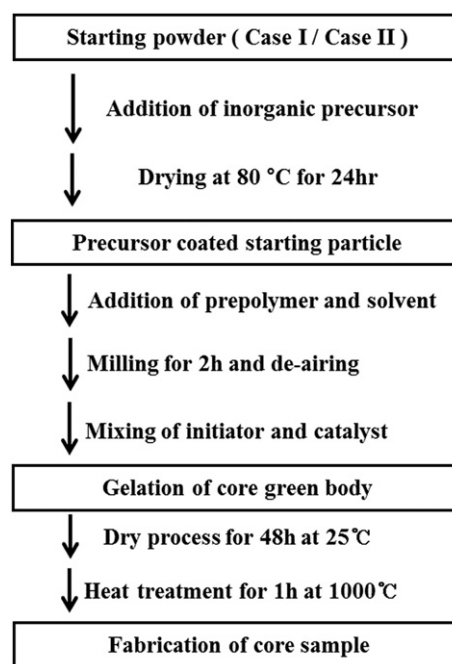


Fig. 1. Schematic diagram of the fabrication of core samples using the gel-casting method and an inorganic precursor.

the temperature was higher than in Case I (Fig. 2(a)). The gelation is directly related to the network structure and polymer density. As a result, the core prepared in Case II would probably have a higher green strength compared with the core prepared in Case I.

The reaction mechanisms involving the two inorganic precursors used in this study, silicate and sodium methoxide, are as follows [8,9]:

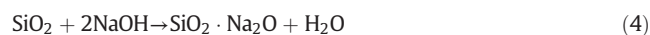
Sol–gel reaction:



Hydrolysis reaction:



Heat treatment:



where $\text{Si}(\text{OEt})_4$, $\text{Si}(\text{OH})_4$, EtOH , SiO_2 , NaOMe , NaOH , and MeOH denote TEOS, silanol, ethyl alcohol, silica, sodium methoxide, sodium hydroxide, and methyl alcohol, respectively [10,11]. Sodium methoxide is hydrolyzed to form sodium hydroxide, and TEOS is converted to silica and ethyl alcohol. The SiO_2 and NaOH synthesized via the above mechanisms are classified to form sodium silicate ($\text{SiO}_2 \cdot \text{Na}_2\text{O}$) during heat treatment. Specifically, liquid-phase precursors are changed to solid-phase glass via the sol–gel reaction and heat treatment, subsequently leading to an improvement in the sintering strength of the core.

Table 2
Formulations and conditions used to prepare core samples using the gel-casting method and an inorganic precursor.

Run number	Inorganic precursor	Starting powder	Additive	Condition
Run (a-1)	TEOS: 38 g NaOMe: 56 g	Case I	60 vol.%	Monomer: 3.3 wt.%
Run (a-2)				Dimer: 0.7 wt.%
Run (b-1)		Case II		Dispersant: 1.1 wt.%
Run (b-2)				Solvent: 40 vol.%
				40 vol.%
			Initiator: 0.15 g	Before heat treatment
			Catalyst: 0.15 g	After heat treatment

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