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Development of a new process in high functioning ceramic core without shape deformation

Eun-Hee Kim[a,*], Geun-Ho Cho[a], YoungSoo Yoo[b], SeongMoon Seo[b], Yeon-Gil Jung[a,**]

[a]School of Nano & Advanced Materials Engineering, Changwon National University, Changwon, Kyungnam 641-773, Republic of Korea
[b]High Temperature Materials Research Group, Korea Institute of Materials Science, Changwon, Kyungnam 641-831, Republic of Korea

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Abstract

A new process was developed to prepare a ceramic core with reasonable strength, and without shrinkage and shape deformation. In this work, a mixture of inorganic precursor composed of silicate and metal alkoxide was used to increase the strength of the core during the wax injection under about 50 °C and the casting processes under about 1300–1350 °C. In the case of the core prepared through the new process, the nominal fracture strength is 12 MPa because of the glass phase synthesized by the inorganic precursor. However, in the conventional process, the core shows the strength of about 6 MPa, as manifested by the sintering effect between starting particles occurring during heat treatment. In addition, the core sample prepared using the inorganic precursor is completely eluted in a sodium hydroxide solution, indicating that the new core process is reasonable for the fabrication of a ceramic core having high strength and crushability.

Keywords: C. Strength; Ceramic core; Inorganic precursor; Glass phase; Crushability

1. Introduction

Conventionally, the ceramic core used for the investment casting of hollow components has been prepared using a mixture of organic compounds (wax and surfactants) and starting powders based on zircon flour (ZrO_2) and fused silica (SiO_2) with various particle sizes [1–4]. Therefore, sintering at high temperature is an essential process to provide strength to the ceramic core. However, heat treatment leads to distortion by shrinkage and produces pores by evaporation of the organic compound of the inner core, resulting in destruction of the core during the casting and a defective product. In addition, the ceramic core should be easily collapsed after casting. Therefore, many studies have been performed to prepare the ceramic core with appropriate fracture strength, as well as without deformation of the shape [5–8]. However, the ceramic core is significantly affected by the size of starting powders, the compound ratio, process parameters, etc. In this work, an inorganic precursor has been used to prepare a ceramic core of optimum strength without shrinkage or shape deformation. The fracture strength in a ceramic core prepared with an inorganic precursor is due to the glass phase generated on the interface and surface of the starting particles rather than the sintering [9]. In addition, two types of process are analyzed to investigate the coating effect of the inorganic precursor [9,10]. In the first process, the green body that is formed with a mixture of the starting powder and organic binder is dipped in the inorganic precursor solution. In the second process, it is prepared with inorganic precursor-coated starting powder and organic binder. The effects of various conditions, such as the size of starting powder and the type of process, on the fracture strength are discussed in detail.

2. Experimental procedure

Commercial garde fused silica, zircon flour, and silica with various particle sizes were used as starting ceramic powders to

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*Corresponding author. Tel.: +82 55 213 2742; fax: +82 55 262 6486.
**Corresponding author. Tel.: +82 55 213 3712; fax: +82 55 262 6486.
E-mail addresses: udam99@changwon.ac.kr (E.-H. Kim), jungyg@changwon.ac.kr (Y.-G. Jung).

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prepare the ceramic core. Compositions of the starting powders with different particle sizes are shown in Table 1. The inorganic precursor was composed of tetraethyl orthosilicate (TEOS, Sigma-Aldrich Korea, Yongin, Korea) and sodium methoxide (NaOMe, Sigma-Aldrich Korea, Yongin, Korea) as the SiO_2 and Na_2O precursors, respectively, as presented in Table 2. In addition, poly(vinyl alcohol) (PVA, Sigma-Aldrich Korea, Yongin, Korea) was used as the organic binder to fix the shape for the green body of the ceramic core sample. In this work, two types of process were considered (see Fig. 1). In the first process (process I), the green body was formed with a mixture of the starting powder and organic binder, and then it was dipped in the inorganic precursor solution. The precursor-coated green body was dried and heat-treated. In the second process (process II), in advance, the starting powder was coated with inorganic precursor, and then dried. The green body was prepared with precursor-coated starting powder and organic binder and heat-treated. The dry process and heat treatment were conducted at 80 °C for 1 h and at 1000 °C for 1 h, respectively. The green body was formed at a pressure of 60 MPa with a cuboid shape of 10 mm × 10 mm × 50 mm, followed by cool isostatic pressing (CIP) at 200 MPa. Formulations used to prepare the core samples with different conditions are shown in Table 3.

Table 1
Compositions of starting ceramic powder with different sizes.

Starting powder	Case I (wt%)	Case II (wt%)	Case III (wt%)
Zircon flour 1	10	10	10
Zircon flour 2	10	10	10
Fused silica	–	10	–
Silica powder (2 μm)	10	10	10
Silica powder (45 μm)	30	25	25
Silica powder (149 μm)	30	25	25
Silica powder (100–200 μm)	10	10	10
Silica powder (200–500 μm)	–	–	10

Table 2
Composition ratio of inorganic precursor.

TEOS (wt% (mol%))	NaOMe (wt% (mol%))	Isobutyl alcohol (wt% (mol%))
38 (0.18)	56 (1.5)	6 (0.08)

The fracture 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 bending mode, at a rate of 0.5 mm min^{-1}. Tests were carried out at room temperature. Ten runs were performed to determine the standard deviation of the fracture strength. The elution test was conducted with 40 wt% NaOH solution at 30 °C for 3 h, using the ceramic core sample after fracture strength test.

3. Results and discussion

The reaction mechanisms involving the two inorganic precursors used in this work, silicate and sodium methoxide, are as follows [11,12]:

Sol–gel reaction:

$$Si(OEt)_4+4H_2O \rightarrow Si(OH)_4+4EtOH \quad (1)$$

$$Si(OH)_4 \rightarrow SiO_2+2H_2O \quad (2)$$

Hydrolysis reaction:

$$NaOMe+H_2O \rightarrow NaOH+MeOH \quad (3)$$

Heat treatment:

$$SiO_2+2NaOH \rightarrow SiO_2 \cdot Na_2O+H_2O \quad (4)$$

where $Si(OEt)_4$, $Si(OH)_4$, EtOH, SiO_2, NaOMe, NaOH, and MeOH denote TEOS, silanol, ethyl alcohol, silica, sodium methoxide, sodium hydroxide, and methyl alcohol, respectively [13,14]. TEOS is degraded to silanol and ethyl alcohol through the hydrolysis reaction (Eq. (1)), and silica is subsequently formed by the condensation reaction between silanol molecules (Eq. (2)). This series of reactions is usually called a sol–gel

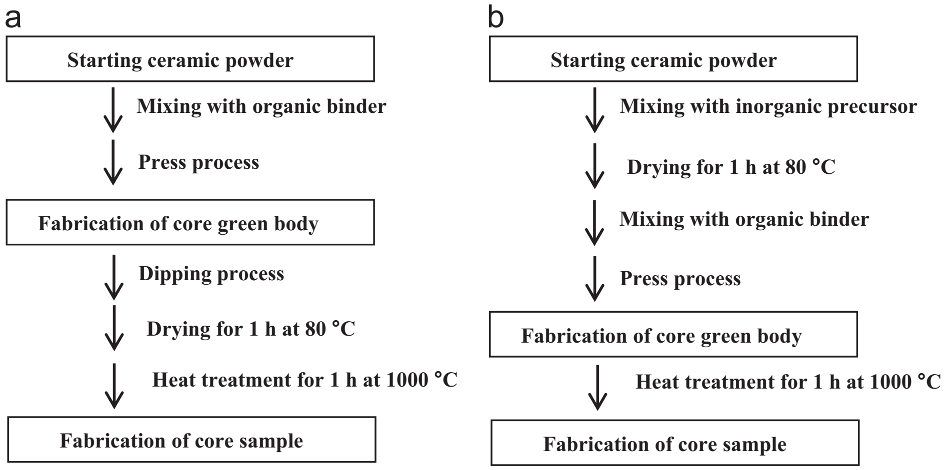

Fig. 1. Schematic diagram for preparing ceramic core samples: (a) process I and (b) process II.

Table 3
Formulations used to prepare ceramic core samples with various conditions.

Run number	Type of starting particle	Organic binder (wt%)	Process	
Run 1-1	Case I	10	Process I	before heat treatment
Run 1-2				after heat treatment
Run 2-1	Case II			before heat treatment
Run 2-2				after heat treatment
Run 3-1	Case III			before heat treatment
Run 3-2				after heat treatment
Run 4-1	Case II	5		before heat treatment
Run 4-2				after heat treatment
Run 5-1		15		before heat treatment
Run 5-2				after heat treatment
Run 6-1		10	Conventional process	before heat treatment
Run 6-2				after heat treatment
Run 7-1			Process II	before heat treatment
Run 7-2				after heat treatment

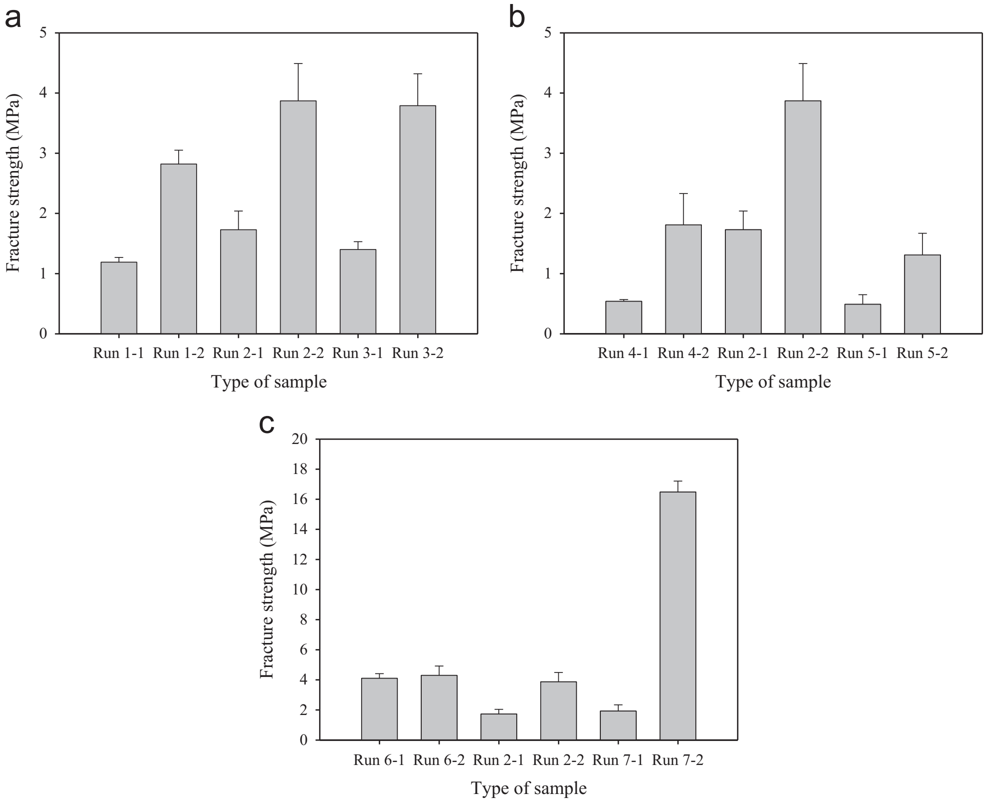

Fig. 2. Fracture strengths of ceramic core samples prepared with different conditions: (a) addition ratio and size of silica particles, (b) content of organic binder, and (c) type of process.

reaction [11,15]. NaOMe is hydrolyzed to form NaOH (Eq. (3)). Then, the SiO_2 and NaOH synthesized via the above mechanisms are glassified to form sodium silicate ($SiO_2 \cdot Na_2O$) during heat treatment. The liquid-phase precursor is converted to a solid-phase glass by the sol–gel reaction and heat treatment, subsequently improving the mechanical properties of the ceramic core. Therefore, the inorganic precursor should be uniformly coated on the surface of the starting powder. Consequently, in this work, the ceramic cores have been prepared through two types of process to maximize the coating effect of the inorganic precursors.

The ceramic core was prepared under various conditions, with varied parameters such as the addition ratio and size of the silica particles, the content of organic binder, and the type of process used to form the ceramic core with optimal properties. Table 1 shows the compositions in the starting powders used in this work. The difference between cases I and III is the addition ratio and size of silica particles, and fused silica is added only in case II. Runs 1−5 and Run 7 were conducted through process I with a dipping process and process II with a coating process, respectively. In general, small particles are added to enhance the sintering effect between particles by the high packing factor, affecting the fracture strength of the core. In process I, the inorganic precursor would migrate into the inner core by capillary phenomena during dipping. Then, the precursor wetted on the surface of the particle is glassified by gelation and heat treatment. In Fig. 2(a), the core sample prepared by case III shows the higher fracture strength than that for case I with the powders of relatively small size. This means that the addition ratio of the silica particles deciding the space between the particles is a more important factor than particles of small size to increase the strength of the core. In addition, the core prepared with case II has the highest fracture strength caused by the increase in the glass phase by fused silica of high purity added to the starting powder.

The adhesion phenomena of inorganic precursors depending on the content of organic binder are shown in Fig. 2(b). After heat treatment at 1000 °C, the strength of the ceramic core is produced solely from the glass phase that occurred on the particle surface because the organic binder used to fix and form the green body is completely burned at the low temperature of about 400 °C. This indicates that the glass phase is uniformly and homogeneously coated on the surface of the particles. The core samples prepared with the optimum content of organic binder show the highest fracture strength (Run 2). This may be due to enhancement of the adhesion force in the glass phase formed between the starting particles. However, too little or too much a content of organic binder has a negative impact on the formation of an ideal interface between the particles. In the former case (Run 4), the formation of the core sample at the given pressure is difficult because of the inadequate amount of organic binder. In the latter case (Run 5), too much a content of organic binder obstructs the contact between glass phases coated on the particles. In particular, it is noted that the adhesion force in Run 2 is higher than those in Runs 4 and 5, producing an enhancement of the fracture strength. In addition, similar patterns are also observed in the core samples before heat treatment.

In this work, two types of process were developed to investigate the coating effect of the inorganic precursor. In process I, the green body consisting of starting powder and organic binder was dipped into the inorganic precursor. The precursor solution moves along the small gaps between the particles by capillary force. However, in the case of process II, the green body was formed with precursor-coated particles. It could be expected that the coating effect of the inorganic precursor is higher for process II than for process I. The strength of the core sample prepared using process II has a very high value, compared with those from samples prepared though the conventional process and process I, as seen in Fig. 2(c). In elution test, the core sample prepared using Run 7-2, which has the highest fracture strength, after fracture strength test was used. The core sample is completely crushed in a sodium hydroxide (NaOH) solution (see Fig. 3). Consequently, the condition introduced in this study, such as case II, 10 wt% organic binder, and process II, can be applied for preparing ceramic cores having high strength and crushability.

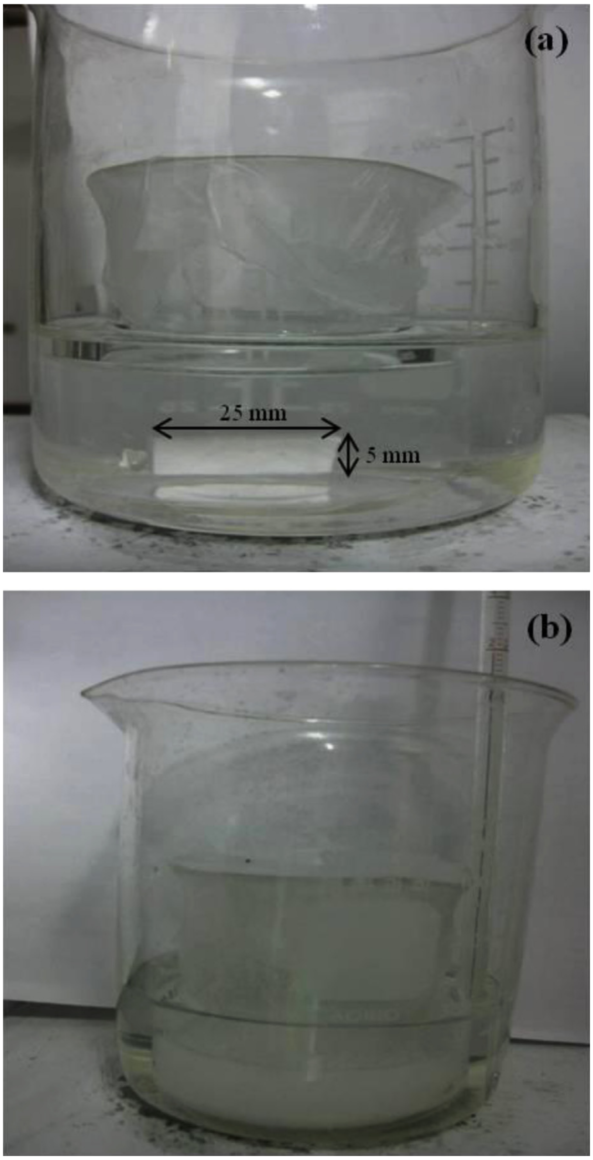

Fig. 3. Elution results of ceramic core sample prepared through process II after heat treatment (Run 7-2) in 40 wt% NaOH solution: (a) before and (b) after collapse. The black arrow indicates the core sample before elution test.

4. Conclusions

In this study, the addition ratio and size of silica particles, the content of organic binder, and the type of process were controlled to prepare a ceramic core with reasonable fracture strength and crushability, and without shrinkage and shape deformation. In particular, a new process was developed to investigate the coating effect of the inorganic precursor. The inorganic and organic binders employed in this study were TEOS and NaOMe, and PVA, respectively. The core sample prepared with the optimum content of organic binder and the proper addition ratio and size of silica particles shows the highest fracture strength, which may be due to the high contact area and uniform development of the glass phase between the particles, resulting in enhancement of the adhesion force. However, in the cases of too little or too much a content of organic binder, the strength of the ceramic core is significantly decreased. This is because there is insufficient organic binder to create the network structure of the glass phase in the mold or it is not uniformly developed between the particles. In addition, in the core sample prepared through process II, the fracture strength is considerably improved because of the effective generation of the glass phase between the particles by high coating efficiency. The core sample prepared through the new process is completely degraded in a sodium hydroxide (NaOH) solution.

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