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Investigation on cristobalite crystallization in silica-based ceramic cores for investment casting

A. Kazemi [a,*], M.A. Faghihi-Sani [b], H.R. Alizadeh [c]

[a] *Department of Materials Engineering, Science & Research Branch, Islamic Azad University, Tehran, Iran*
[b] *Department of Materials Science and Engineering, Sharif University of Technology, Tehran, Iran*
[c] *MAPNA Turbine Blade Eng. & Mfg. Co., PARTO, Iran*

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Abstract

In this work, cristobalite crystallization and its effects on mechanical and chemical behaviour of injection moulded silica-based ceramic cores were investigated. In order to simulate casting process condition, the sintered samples at 1220 °C were also heated up to 1430 °C. Flexural strength test was carried out on both sintered and heat treated samples. Chemical resistance of the cores was evaluated by leaching the samples inside 43 wt% KOH solution at its boiling point. Phase evolution and microstructure were investigated by thermal analyses (DTA and DSC), X-ray diffraction (XRD), scanning electron microscopy (SEM) and optical microscopy (OM). Results showed that cristobalite was crystallized on the surface of fused silica grains at about 1380 °C. Flexural strength of the sintered cores was decreased after simulated casting heat treatment due to cristobalite phase transformation. The formed cristobalite on the surface of fused silica grains dramatically decreased the leachability of ceramic cores.

Keywords: In situ cristobalite; Crystallization; Leachability; Fused silica; Ceramic core

1. Introduction

Ceramic cores with very precise and often complex shapes are manufactured by injection moulding to achieve a near net shape. They are extensively used in turbine blade casting. Proper match between the ceramic core, investment casting ceramic mould and metal blade is essential for the proper performance of the casting process.[1] Silica-based ceramic cores are extensively used for casting nickel based blades with casting temperature of lower than 1500 °C due to their high dimensional stability, thermal shock resistance, and leachability.[2] These cores are usually composed of silica, zircon, and negligible amounts of cristobalite and alumina.[3,4] The β-cristobalite, as a high temperature polymorph of silica, undergoes β → α phase transformation with a volume contraction of about 5 vol% when it is cooled down to 200–270 °C.[5,6]

In this study, crystallization of fused silica and its effects on the most important properties of injection moulded silica-based ceramic cores, including flexural strength and leachability, have been investigated.

2. Experimental procedure

The silica-based ceramic cores containing 65.2 wt% fused silica (Remet, purity 97.3%; $d_{50} = 27.7$ μm), 16 wt% zircon (Cookson, purity 88%; $d_{50} = 20.4$ μm), 2.8 wt% cristobalite (Sibelco, purity 99.4%; $d_{50} = 14.1$ μm) and 1.2 wt% alumina (Ceramatco, purity 96.9%; $d_{50} = 9.4$ μm) were fabricated through injection moulding.

To investigate crystallization temperature of fused silica in the prepared ceramic cores, Differential Thermal Analysis (DTA) was carried out on a 7 mm × 7 mm × 5 mm *LWH* sample from room temperature up to 1430 °C with a heating rate of 10 °C min^{-1}. In addition, differential scanning calorimetry (DSC) analysis was also conducted to confirm in situ cristobalite formation. The fused silica powders with various grain sizes (as shown in Table 1) were also heated at various temperatures (1200, 1300 and 1400 °C) for 1 h and then were analyzed

* Corresponding author. Tel.: +98 912 225 0115; fax: +98 2144869783.
E-mail addresses: arg.kazemi@gmail.com, a_cercis@yahoo.com, a.kazemi@srbiau.ac.ir (A. Kazemi).

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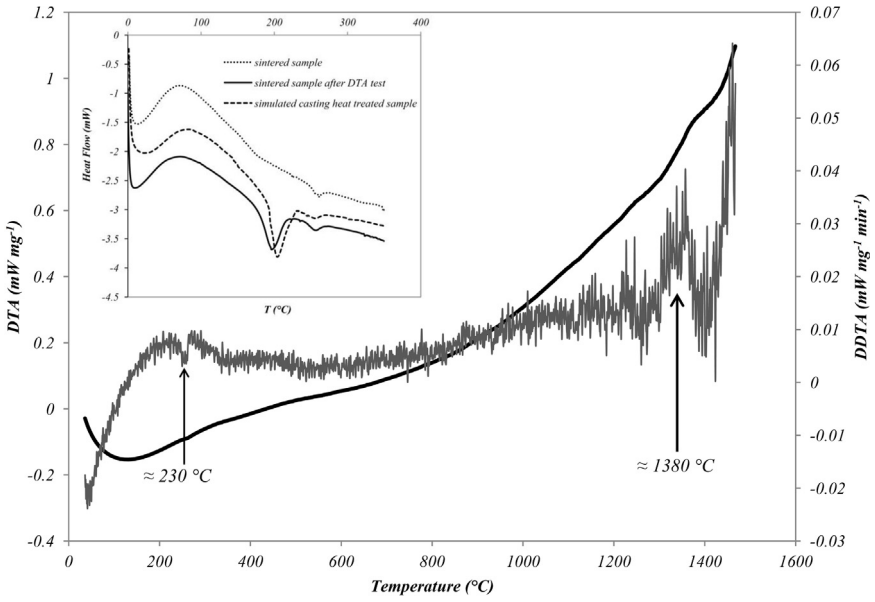

Fig. 1. DTA and DDTA graphs of 7 mm × 7 mm× 5 mm ceramic core from 25 to 1430 °C as well as DSC results of the sintered sample, the sintered sample after DTA test and the simulated casting heat treated sample (all with heating rate of 10 °C min^{-1}).

by an X-ray powder diffractometer with Ni-filtered CuKα radiation, (PW1800-Philips) with 0.04 step size and 4 s detecting time, in order to study the fused silica crystallization. Polished cross sections and fractured surfaces of the prepared samples were also monitored by optical microscope (Olympus BH-2) and scanning electron microscope (SEM, VEGA TESCAN, Czech, 15 kV).

To study the effects of alumina addition on fused silica crystallization, two series of compacts containing fused silica particles with and without 10 wt% fine alumina particles were sintered at 1220 °C for 6 h, and some of them were subsequently heat treated at 1400 °C for 1 h. For semi-quantitative analysis, 10 wt% zircon powder was added to the heat treated samples before XRD test. Then, intensity ratio of cristobalite peak (1 0 1) to zircon peak (2 0 0) was calculated for each sample.

In order to investigate the effect of in situ formed cristobalite on mechanical and chemical behaviour of the silica-based ceramic core, the injection moulded test bars of 150 mm × 22 mm × 5 mm *LWH* were first sintered at 1220 °C for 6 h, and were subsequently subjected to a simulated casting heat treatment at 1430 °C for 30 min. Then, flexural strengths of the sintered samples, before and after heat treatment, were measured by a three-point bending test (SANTAM STM-5 machine with span distance of 100 mm and 6 N s^{-1} loading rate) according to ASTM C674. Each flexural strength result was an average of five bending tests.

Table 1
Specifications of the used fused silica powders.

Powder	PSD			Specific surface area (m^2 g^{-1})
	D_{10} (μm)	D_{50} (μm)	D_{90} (μm)	
Fused silica (1 0 0)	4.37	49.6	177.36	0.513
Fused silica (2 3 0)	2.74	27.7	105	0.777
Fused silica (4 0 0)	1.45	6.58	79.73	1.830

The sintered specimens, before and after simulated casting heat treatment, were cut into 22 mm × 10 mm × 5 mm *LWH* pieces and were leached inside a 43% KOH solution at its boiling point for 15 and 30 min, respectively. Then, leaching rate was calculated according to Eq. (1), where ΔW was the total weight loss (g), A was the initial surface area (cm^2), t was the leaching time (h), and ρ was the specimen density (g cm^{-3}).

$$K = \frac{\Delta W}{A\rho t^{1/2}} \quad (1)$$

This equation provides leaching rate independently of the sample size and shape, which fits well with diffusion controlled model for leaching process.[7]

3. Result and discussion

3.1. Cristobalite crystallization

The derived differential thermal analysis (DDTA) graph of the sintered ceramic core at 1220 °C in Fig. 1 showed two peaks at 230 and 1380 °C. The first one indicates α → β polymorphic transformation of primary cristobalite, and the second one is related to the crystallization of cristobalite.

DSC thermal analyses of the sintered sample, the sintered sample after DTA test (heated up to 1430 °C with a rate of 10 °C min^{-1}), and the simulated casting heat treated sample, in Fig. 1, also show that intensity of the cristobalite polymorphic transformation peak (around 200 °C) increases after the DTA test or after the simulated casting heat treatment, demonstrating