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Effects of Alumina on Cristobalite Crystallization and Properties of Silica-Based Ceramic Cores

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Key words: Silica-based ceramic cores Alumina Cristobalite crystallization Properties In this work, the influences of alumina addition on cristobalite crystallization and properties of injection molded silica-based ceramic cores were investigated. X-ray diffraction (XRD) was used to characterize phase transformations in the samples, and the XRD result indicated that the addition of alumina promoted crystallization of fused silica during sintering at 1180–1220 °C and thus increases the amount of cristobalite. The increased amount of cristobalite as well as alumina addition led to much more thermal dilation due to their higher coefficients of thermal expansion than that of fused silica. The flexural strengths at room temperature and 1500 °C were tested, and it was shown that alumina addition could not affect room temperature strength, but decreased the flexural strength at 1500 °C. In addition, deflection resistance during heating to high temperatures was investigated, and the result indicated that alumina addition speeded up high temperature softening of the samples. XRD and scanning electron microscopy equipped with energy dispersive spectrometry (SEM/EDS) analysis suggested that this softening behavior was related with viscous flow sintering which could be accelerated by the reaction of alumina and silica with a product of mullite.

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1. Introduction

Fused-silica based ceramics are often used as core materials in precise investment castings to produce hollow cooling passages within the metal product, e.g., superalloy turbine blades and vanes^[1–3]. In order to produce acceptable inner surface finish and thickness of the wall for the blades or vanes, the properties of the ceramic cores have to be balanced carefully, such as mechanical strength, thermal stability, dimensional conformity, thermal shock resistance, chemical inertness against superalloys, enough mechanical weakness during solidification, and easy removability by a process that is not harmful to the metal^[4–7]. Many studies have been conducted to prepare the ceramic cores with appropriate performance.

It has been proved that crystallization behavior of fused silica (fused silica \rightarrow cristobalite) in the silica based ceramics has a crucial effect on their performance. The relationship between the cristobalite crystallization and mechanical strength has been investigated previously^[8], and the results indicate that the core materials with

* Corresponding author. Ph.D.; Tel.: +86 24 23971787; Fax: +86 24 23971758. *E-mail addresses: jjliang@imr.ac.cn* (J.J. Liang): yzzhou@imr.ac.cn (Y.Z. Zhou). a small quantity of cristobalite show poor refractoriness and cause excessive bending during casting. Some researchers^[9,10] suggest that the core strength decreases when the content of cristobalite is higher than 16%. Kato and Nozaki^[11] reported that the silica based material containing 10–20 wt% cristobalite could be applied for superalloy casting, and in this case, softening and shrinkage of the core at high temperatures could be restrained.

MATERIALS SCIENCE &

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Since the amount of cristobalite plays a vital role in determining the performance of the cores, it is necessary to study the factors that influence the cristobalite crystallization. Wang and Hon^[7] studied the transformation kinetics of the fused silica with and without cristobalite seed by quantitative X-ray diffraction (XRD) analysis, and their results indicated that cristobalite seed assisted crystallization of fused silica at the initial stage of heating, but retarded crystallization as soaking time went on. Kazemi et al.^[6] and Wilson et al.^[12] suggested that the addition of zircon could prevent cristobalite crystallization by introducing a pinning force at the grain boundaries. Kazemi et al.^[5] also investigated the effect of alumina addition on the crystallization of fused silica, but in their study, zircon was added into the cores as well as alumina.

From the available studies, it is seen that the effects of the sole alumina addition on the cristobalite crystallization and mechanical properties of silica based ceramics have not been investigated.

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2

ARTICLE IN PRESS

J.J. Liang et al. / Journal of Materials Science & Technology ■■ (2016) ■■–■■

Table 1

Weight percentages of compounds in the raw materials (wt%)

	SiO ₂	Al_2O_3	Fe ₂ O ₃	TiO ₂	MgO	Na ₂ O	K ₂ O
Fused silica Alumina	99.87 0.028	0.017 99.66	0.004 0.0096	0.002 0.0023	0.001 0.0008	0.015 0.081	0.004 0.0002

Therefore, in the present study, the crystallization behavior and mechanical properties, e.g. flexural strength and deflection, of silica based ceramics with different alumina additions (5, 15, and 25 wt%) were investigated.

2. Experimental

The raw materials in the present experiment are commercially available ceramic powders (fused silica and fused corundum), and their chemical components are listed in Table 1. The particle size of the fused corundum is ~5 μ m. The purchased fused silica was ball milled first and then sieved to obtain ~300 mesh particles. The particle size distribution of the obtained silica particles is shown in Fig. 1. Three kinds of ceramic bar samples (100 mm × 10 mm × 4 mm) with different alumina additions (5, 15, and 25 wt%), denoted as CKB1, CKB2, and CKB3, respectively, were fabricated through injection molding method. These prepared samples were then sintered at electrical resistance furnace. During sintering, the samples were heated up to 450 °C at a heating rate of 80 °C/h, and holding 4 h at this temperature for dewaxing. Then, heating continued at a heating rate of 120 °C/h until the temperature reached 1180, 1200 and 1220 °C.



Fig. 1. Particle size distribution of the materials used in this work.

The approximate quantities of phases in the investigated samples were identified by an X-ray diffractometer with Ni-filtered CuK α radiation (XRD, D8 Discover, Bruker, Germany). Microstructures and phase compositions were characterized by scanning electron microscopy equipped with energy dispersive spectrometer (SEM/ EDS. ISM-6301F. IEOL. Japan). Thermal expansion behavior was characterized by a UnithermTM-1252 ultra high temperature dilatometer with an error of $< \pm 5\%$ between 200 and 500 °C and $<\pm 4\%$ between 500 and 2500 °C. The heating rate during thermal expansion testing was 5 °C/min. The sample surfaces that were subjected to the anvil of the equipment were protected by slices of boron nitride (BN). The mechanical shock resistance against waxing and molten pouring was characterized by flexural strength. The flexural strengths were tested at room temperature and 1500 °C through a three point bending test. The span distance of the samples during this bending test was 100 mm. Samples that would be tested at 1500 °C were heated at a rate of 10 °C/min and held at 1500 °C for 30 min before testing. Probe that carried force was then loaded at a rate of 1 mm/min until these tested samples were fractured. Dimensional/shape stability during thermal process was estimated through deflection test. Fig. 2 shows the schematic diagram of the deflection test. One end of the sample is fixed, and the other end is hanged (Fig. 2(a)). The sample was then heated to 1600 °C at 10 °C/min and held at 1600 °C. During this thermal process, the sample deformed under its self-weight, and its hanged end drooped gradually. The drooping distance (Δh) of this hanged end was recorded (Fig. 2(b)), and was used to characterize its dimensional/ shape stability.

3. Results and Discussion

3.1. Cristobalite crystallization

It has been proved that during sintering, crystallization of the fused silica always occurs on the surface of the fused silica grains^[5,6,12]. So in the present study, in order to characterize the surface microstructure of the ceramic grains, the samples were intended to be ruptured at room temperature through a three point bending.

SEM microstructures of these samples that have been sintered at different temperatures are shown in Fig. 3. It is seen from these figures that both the grains of the silica and alumina display irregularly sharp-edged shape, and the cavities among the grains have irregular appearance too. All the samples display this microstructure morphology no matter what the alumina content and sintering temperature are. XRD patterns of the samples, shown in Fig. 4, indicate that cristobalite peaks are presented in all the samples. This confirms that crystallization of fused silica (fused silica \rightarrow cristobalite



Fig. 2. Schematic diagram of sample position in deflection test: (a) original position and (b) drooping distance of the hanged end.

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