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Full Length Article

Effect of silica fiber on the mechanical and chemical behavior of alumina-based ceramic core material

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ABSTRACT

In order to improve the chemical leachability, the alumina-based ceramic core material with the silica fiber was injected and sintered at 1100 °C/4 h, 1200 °C/4 h, 1300 °C/4 h and 1400 °C/4 h, respectively. The micrographs of ceramic core materials at sintered and leached state were characterized by scanning electron microscopy (SEM). The phase composition of ceramic core material after sintering and the leaching product after leaching were detected by X-ray diffraction (XRD). The porosity, room temperature bend strength, creep property at elevated temperature and the leaching rate in aqueous caustic solution were studied. The experimental results show that the ceramic core material with silica fiber obtain a fair balance between mechanical and chemical properties at sintering of 1300 °C/4 h. Specifically, the leach rate of ceramic core material with silica fiber is increased apparently. High leaching surface and weak adhesive strength between agglomerated alumina particles are the reasons that responsible for the ceramic core material with silica fiber be leaching fast than that of the ceramic core material without fiber.

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1. Introduction 21

Directionally solidified and single-crystal superalloys have been 22 employed to increase the gas turbine entry temperature [1]. Up to 23 date, the supealloys have been used near the melting temperature. 24 25 Therefore, cooling technique was widely used in all the investment casting advanced hollow turbine blade to endure the elevated gas 26 temperature and this is resulting in a very complex internal cool-27 ing channel of the turbine blade. Ceramic core which used forming 28 internal cooling structure plays an essential role during the cast-29 ing hollow turbine blade. Usually, the ceramic core is quite thin 30 in cross-section typically and may include complex details to form 31 narrow cooling channels, internal support ribs and other features. 32 In addition, the ceramic core must be physically and chemically 33 stable at elevated temperatures, enough strength to be resistant to 34 thermal shock, sufficiently resistant to dimensional changes, non-35 reactive to the superalloy casting, and leachable from the turbine 36 blade within a commercially reasonable time. As a result, the crucial 37 point during producing the gas cooling turbine blade is to fabricate 38 the high property ceramic core [2-6]. 39

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Fused silica-based ceramic core have been widely used to fabricate the investment casting hollow turbine blade. However, this kind of ceramic core may react with the elements of C, Ti, Al in supealloys at elevated temperature and form casting defects, such as metal oxide, gas porosity and burnt-on, and those defects will decrease the use temperature of the turbine blade [7,8].

The other widely used ceramic core is alumina-based ceramic core, which has many advantages, such as its equivalent thermal expansion coefficient to corundum shell, its excellent chemical stability and high temperature properties. However, alumina is very difficult to leach out from the inner of the turbine blade by alkali and acid, and this disadvantage limit its application in directional solidification of superalloy turbine blade [9,10].

Many works have been done to improve the alumina-based ceramic core material overall properties [8–13]. For examples: Wu et al. [8] improved the high temperature properties of aluminabased ceramic core by adding yttria in vacuum impregnating method. The experimental results showed that aqueous yttria sol can improve the high temperature properties of ceramic cores [8]. Qin and Pan [10] investigated the effect of colloidal silica sol on the high temperature properties of alumina-based ceramic core material, and it was found that the ceramic cores with 5 wt.% colloidal silica sol had the smallest creep deformation and the lowest thermal expansion [10]. Bin et al. [12] found that the reasonable **04** 63 particle size match of coarser, middle and finer matrix alumina will

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be beneficed for properties of ceramic core [12]. Lee et al. [13] displayed that the addition of the alumina fiber can strengthen the alumina composites [13]. However, almost all the above mentioned works only focus on improving the mechanical properties, especially the strength and the creep resistance of the alumina-based ceramic core material. Few of works have been done to improve 70 the leachability of the alumina-based core material. Generally, the 71 higher the porosity is, the better the leachability is. However, the 72 high porosity will deteriorate the mechanical properties of the 73 alumina-based ceramic core materials. So it is a challenged works to 74 improve the leachability and keep the high mechanical properties of the alumina-based ceramic core material in the meantime. 76

In this paper, alumina-based ceramic core material with silica fiber was prepared. The micrographs of ceramic core materials at sintering and leached state were characterized by SEM. The porosity, bend strength at room temperature, the creep resistance at 80 high temperature and the leaching rate in aqueous caustic solution were studied. The effect of silica fiber on the leachability of 82 alumina-based ceramic core material was discussed.

2. Experimental details 84

The alumina powder, silica powder and silica fiber in the present experiment is commercially provided. The alumina powder (supplied by Shandong Zibo Aluminum Inc. China) was consisted of coarse particles (d_{50} is 75 μ m) and fine particles (d_{50} is 10 μ m), and the ratio of coarse particles to fine particles is 30:70 (mass percent). Silica powders was chosen as the mineralizers (supplied by Jiangsu Jinhao Co., Ltd. China) and the d₅₀ of silica powder is 5 µm. The silica fiber was chemically pured (provided by Wuhan Technophile Co., Ltd. China) and the diameter of the fiber is $8-10 \,\mu m$ and the length is 1-2 mm.

Two kinds of alumina-based ceramic core materials were prepared. One of the ceramic core materials was composed of 99 wt.% alumina powder and 1 wt.% silica powder; and the other one is 98 wt.% alumina powder, 1 wt.% silica powder and 1 wt.% silica fiber. Both of two ceramic core powders were ball milled for 10 h.

The micrograph of mixed alumina-silica powder and silica fiber 100 was observed by SEM (S-3400N, Japan). The phase transitions of 102 silica fiber was measured by differential scanning calorimeter (Netzsch DSC 404C) in heating runs. The mass of the silica fiber is 55.50 mg and the scanning rate is 10 K/min. The particle size distribution map of ball milled alumina-silica powders was tested by Laser Particle Size Analyzer (BT-9300Z, China).

After ball milling, two kinds of ceramic core slurry were prepared. which were consisted of mixed powder or mixed powder/fiber and wax binder, respectively. The mass ratio of powder or powder/fiber to wax binder is 85:15. Both two kinds of ceramic core materials were injected in dies at 5 MPa and different size green ceramic samples were prepared.

These green ceramic samples were sintered at 1100°C/4h, 1200 °C/4h, 1300 °C/4h and 1400 °C/4h, respectively. After sintering, the samples were cooled with furnace. The micrograph of ceramic core materials after sintering was observed by SEM and the phase composition was analyzed by XRD. The porosity of sintered ceramic core materials was measured by using the Archimedes principle, in which water as the immersion medium.

Rectangular sintered bars with dimension 120 $60 \text{ mm} \times 10 \text{ mm} \times 4 \text{ mm}$ were prepared for bend strength testing 121 through three point bend method. At least five samples were 122 tested and the average value was taken as the testing result. 123

Rectangular sintered bars with dimension 124 of $2 \text{ mm} \times 6 \text{ mm} \times 120 \text{ mm}$ were prepared for the creep prop-125 126 erty testing. The creep deformation of the sintered alumina-based 127 core material was evaluated by double support point test. The length of the span is 100 mm. Each bar was put on the sample setter in a furnace and the heating rate is 600 °C/h and heat to 1550 °C then holding at 1550 °C for half an hour. For each condition five samples were tested and the average value was set as the result

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The sintered bars were leached by 30 wt.% aqueous caustic solution at 160°C. The dimension of rectangular bars is $4 \text{ mm} \times 10 \text{ mm} \times 30 \text{ mm}$. The weight loss rate is expressed as $C = M/(t \times S)$, where C, M, t and S are leaching rate, the mass loss of the alumina-based ceramic sample, leaching time and original surface area of the sample, respectively. In our experiment, the leaching time is 10 h.

In order to observe the leaching surface micrograph, the typical alumina ceramic core materials with and without silica fiber sintered at 1300 °C/4 h were leached by aqueous 30 wt.% caustic Q5 solution at 160°C for five hours and the leaching product was detected by XRD. For understanding the leaching process of silica fiber, the ceramic core materials same as above were leached by aqueous hydrofluoric acid (30 wt.%) solution at room temperature for five hours and the micrograph of the leaching material surface were observed by SEM.

To compare the leaching macro morphology, these two kinds of ceramic core materials sintered at 1300 °C/4 h were leached in aqueous hydrofluoric acid (30 wt.%) at room temperature after 20 h were interrupted.

3. Results and discussion

The diameter of the fiber is about 10 µm, the length of the fiber is at millimeter class and there is no micro crack on the surface of the fiber. The DSC curve of silica fiber is shown in Fig. 1. The nucleation and structure rearrangement take place on the surface of the silica fiber at 1153.95 °C. The crystallization starts at 1336.46 °C and end at 1398.96 °C. The micrograph and size distribution graph of mixed alumina-silica powder after milling is shown in Fig. 2. The mixed powder is consisted of coarse particles and fine particles (Fig. 2(b)). Usually, coarse particle in ceramic core material is benefit to anti creep deformation at elevated temperatures, and fine particles is benefit to improve the bend strength and the surface finish of the ceramic core.

The micrograph of two kinds of ceramic core materials sintered at different temperature is shown in Fig. 3. It is demonstrated from Fig. 3 that with increasing of the sintering temperature the pore size of the ceramic core materials is continuous decreasing regardless of the core material with and without silica fiber. It is also shown in Fig. 3(f) and (h) that the micro crack formed on the surface of

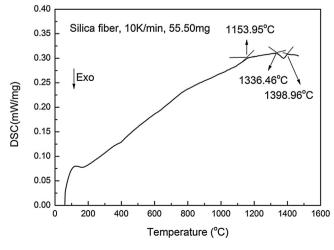


Fig. 1. DSC curve of silica fiber.

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