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### Experimental study on mechanical properties of silica-based ceramic core for directional solidification of single crystal superalloy



Zilin Xu, Jiangwei Zhong, Xianglin Su, Qingyan Xu\*, Baicheng Liu

Key Laboratory for Advanced Materials Processing Technology (MOE), School of Materials Science and Engineering, Tsinghua University, Beijing 100084, China

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#### ABSTRACT

The effects of holding time in Bridgman furnace on mechanical properties of injection molded silica-based ceramic cores for directional solidification of SX superalloy have been investigated. The cylindrical samples (S0) were sintered at 1000 °C for 60 min, and some of the sintered samples (S1, S2, S3) were treated by heat treatment at 1500 °C to simulate the directional solidification process. Isothermal uniaxial compression tests of ceramic core samples were conducted on a Gleeble-1500D mechanical simulator. Weibull approach was used to describe the strength distribution of silica-based ceramic cores. As a result, the mean compressive strength of the sample (S0) is 40.43 MPa. The mean compressive strengths of the samples (S1, S2, S3) with heat treatment at 1500 °C are 54.34 MPa, 53.60 MPa and 53.81 MPa, respectively, which are significantly larger than that of S0. The mean elastic moduli of the samples (S1, S2, S3) with heat treatment at 1500 °C are 2726.39 MPa, 2855.91 MPa and 2797.14 MPa, respectively, which is significantly higher than that of S0. The refractory particle evolution of the ceramic core during the directional solidification process is analyzed, and the microstructural investigations show that the crack propagation of ceramic core sintered at 1000 °C is mainly through the sintering necks between particles. However, the crack propagation of ceramic core holding at 1500 °C is extended through the entire large particles. The re-sintering process of ceramic core holding at 1500 °C compensates the negative effect of cracks due to the volume contraction during  $\beta$ - to  $\alpha$ -phase transformation and the rapid cooling process, and improves the ceramic core uniformity and mechanical properties.

#### 1. Introduction

Single crystal (SX) gas turbine blades are the extremely important components in the gas turbine engine. They have to serve in high temperature and high pressure conditions with a rotating speed of 10000 rpm, as well as withstand a tension of more than 300 MPa. Preventing the failure of SX gas turbine blades requires modern and advanced alloy materials and processing technology, which have been the focus of research recently [1]. The state-of-the-art technologies to manufacture the SX gas turbine blades include the directional solidification, where complexly designed ceramic cores fabricated by injection molding are widely used to form internal cooling passages in the hollow gas turbine blades [2-4]. Since the ceramic core needs undergo large thermal stress at high temperature, it has been manufactured using a mixture of ceramic powders consisting of fused silica (SiO<sub>2</sub>) and zircon (ZrSiO<sub>4</sub>) due to their excellent high temperature properties, such as thermal shock resistance and chemical inertness against molten metal [5]. Fused silica is mainly used to produce refractory materials, as well as ceramic cores, because of its low thermal expansion coefficient

 $(0.55 \times 10^{-6})$ K between 25 °C and 1000 °C) and excellent chemical inertness against molten metal [6].

During the directional solidification process of superalloy, the contact surfaces between the ceramic core and the superalloy are frequently stress-concentrated, which can cause the destruction of the contact surface. Further, the existence of contact stress is the important reasons of recrystallization in SX blades [7,8].

In the current technology of directional solidification, the fracture mechanism of silica-based ceramic cores is not well understood. Wang and Hon studied transformation kinetics of fused silica with and without cristobalite seed. The results showed that the cristobalite seed could induce a compressive stress due to the abrupt volume increase as cristobalite transformed from  $\alpha$  phase to  $\beta$  phase at elevated temperature [0]

Breneman et al. investigated the effect of phase transition of cristobalite on the flexural strength of fused silica [10]. They reported that the cristobalite improved the flexural strength of fused silica at 350 °C because of the existence of  $\beta$ -cristobalite. However, the flexural strength at room temperature was reduced due to microcracks formed

E-mail address: scjxqy@tsinghua.edu.cn (Q. Xu).

<sup>\*</sup> Corresponding author.

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by  $\beta$ - $\alpha$  transition in cristobalite. The volume contraction during the phase change of cristobalite accompanied with microcracking caused a reduction of the flexural strength of the silica-based ceramics [11].

In addition, Wilson et al. reported that increasing the amount of zircon could have beneficial effects on some properties such as creep resistance and flexural strength at 1475 °C. They also reported that silica, with high surface energy, could increase the sintering rate and consequently improve strength/creep resistance of ceramic core. Moreover, they showed that zircon, as an inert secondary phase, could act as a Zener pinning agent at high temperature and prevent consolidation by introducing a pinning force at the grain boundaries [12].

Kazemi et al. Indicated that improvement in sintering compensated the negative effect of in-situ formed cristobalite during the simulated casting heat treating and its further  $\beta$ - to  $\alpha$ -phase transformation during cooling [13]. This result is contrary to the Wang and Hon report [14], but it is in a good agreement with the Wilson et al. result [12].

Therefore, lack of information about mechanical behavior in silicabased ceramic cores and even their contradictory results make us to study the effects of directional solidification mechanical properties of silica-based ceramic cores. During the directional solidification process of the superalloy, the furnace temperature is higher than the transformation temperature of cubic  $\beta$ -cristobalite (about 1470 °C) [15], which is mainly determined by the melting point of the superalloy, (for example, the second-generation superalloy DD6 has a melting point of 1370 °C [16]). Hence, the holding time of ceramic cores in the Bridgman furnace should be further studied during the actual production of casting.

#### 2. Experimental procedure

The Bridgman method is used for the directional solidification of SX blades. The whole process includes melting, pouring and solidification in the directional solidification furnace. The schematic of the furnace is shown in Fig. 1, which mainly includes a heating zone, a graphite baffle, a copper plate, a cooling zone and a withdrawal device [16]. After the superalloy is poured, the superalloy, core and shell with water-cooled copper plate move from the heating zone to the cooling zone, and the ceramic core undergoes a rapid cooling process. During the process the drawing rate is 4 mm/min, the heating zone temperature is set to 1500 °C, and the temperature of circulating cooling water is 40 °C.

Characteristics of the used fused silica and zircon, as raw materials, are illustrated in Table 1. Porous silica-based ceramic cores were prepared by using ceramic injection molding [13]. After a slow heating step to burn out the thermoplastic binders, they were sintered at

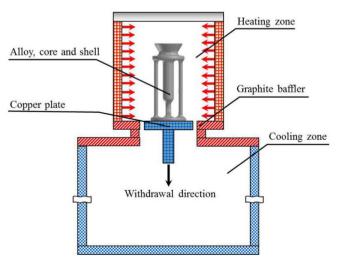


Fig. 1. Schematic of simplified structure of directional solidification furnace.

1000 °C for 60 min using the box type furnace and were defined as S0 samples. For a withdrawal rate of 4 mm/min in the Bridgman directional solidification furnace, the heating zone holding time of ceramic cores in a cast with a total height of 400 mm is more than 90 min. Hence , in order to simulate the realistic directional solidification process, some of the sintered samples (S1, S2, S3) were subsequently subjected to heat treatment at 1500 °C for 30 min, 60 min and 90 min, respectively. The ceramic core samples (S1, S2, S3) were removed from the furnace at 1000 °C to the atmosphere at 25 °C for simulating the realistic rapid cooling process during the directional solidification from the heating zone into the cooling zone. The average size of the ceramic cores is 14.77 mm in diameter and 15.25 mm in length ( $\phi$ 14.77 mm × 15.25 mm). The heat treatment of ceramic core samples to simulate the directional solidification process is summarized in Table 2.

The Gleeble system has been widely used in the research of materials for constitutive laws. It is mainly composed of thermal system, mechanical system and computer control system [17,18]. Cylindrical samples were prepared, and applied in isothermal uniaxial compression tests on a Gleeble-1500D mechanical simulator. The strain-rate of  $10^{-4}$ /s was chosen. Taken into consideration the inhomogeneity of the ceramic core, the test of four samples (S0, S1, S2, S3) repeats 3–6 times. The experimental results of the ceramic core mechanical properties are mainly processed by the arithmetic mean:

$$\overline{M} = \frac{1}{N} \sum_{i=1}^{N} M_i \tag{1}$$

Where N is the number of sample tests, M is critical strain, compressive strength or elastic modulus.

#### 3. Results and discussion

The stress-strain curves of the samples (i.e. S0, S1, S2, S3) are shown in Fig. 2. The elastic-brittle properties are clearly indicated that at small strains the stress-strain behavior is elastic, but if the strain is beyond a critical value, the brittle fracture occurs. The critical strain (about 0.023) of the samples (S1, S2, S3) is larger than that (about 0.017) of the sample S0.

Fig. 3 shows the compressive strength and elastic modulus of samples (S0, S1, S2, S3). As shown in Fig. 3, the compressive strength and the elastic modulus of these samples have a similar trend. The mean compressive strength of the sample (S0) is 40.43 MPa. The mean compressive strengths of the samples (S1, S2, S3) with heat treatment at 1500 °C are 54.34 MPa, 53.60 MPa and 53.81 MPa, respectively, which are significantly larger than that of S0. In addition, with the heat treatment time increase, the mean compressive strengths of samples do not vary much but the standard deviations become smaller, implying a more stabilized property of the sample. The mean elastic moduli of the samples (S1, S2, S3) with heat treatment at 1500 °C are 2726.39 MPa, 2855.91 MPa and 2797.14 MPa, respectively, which is significantly higher than that (2401.91 MPa) of the sample S0.

A common empirical method to describe the strength distribution of a brittle material is the Weibull approach [19,20]. The three-parameter Weibull distribution for the failure of samples at a stress ( $\sigma$ ) can be written as

$$F = 1 - \exp\left[-\int_{V} \left(\frac{\sigma - \sigma_{\min}}{\sigma_{0}}\right)^{m} dV\right]$$
 (2)

Where F is the failure probability, m is the Weibull modulus,  $\sigma_0$  is the characteristic strength and  $\sigma_{\min}$  is the minimum strength. In many cases, a two-parameter Weibull distribution is always assumed, with  $\sigma_{\min}$  equal to 0. Then Eq. (1) can be simplified as follows

$$\ln\left(\frac{1}{1-F}\right) = L_{\rm F} V \left(\frac{\sigma_{\rm max}}{\sigma_0}\right)^m = \left(\frac{\sigma_{\rm max}}{\sigma_0^*}\right)^m \tag{3}$$

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