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An ascending thermal shock study of ceramics: Size effects and the characterization method

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HIGHLIGHTS

- Ascending thermal shock resistance is studied by using an efficient method.
- The effect of specimen size on ascending thermal shock resistance is studied.
- The testing succeeds in developing the internal cracks in the shocked specimen.
- A new characterization method for ascending thermal shock resistance is proposed.
- A simple method for modelling ΔT_C of rectangular specimen is proposed.

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ABSTRACT

The ascending thermal shock resistance of ceramics is studied using a new, simple and efficient test method. The results show that our testing succeeds in developing internal cracks in the shocked specimen that are cause by tensile stresses, while there is no visible change in the surface morphology. This coincides with the failure mechanism of ascending thermal shock, and this has not been reported before. Considering there is no proper experimental characterization parameter for evaluating the ascending thermal shock resistance, we propose an experimental evaluation index called the critical temperature difference of rupture, which is defined as the temperature difference after which a sharp decrease of retained strength appears until fracture. The value of the critical temperature difference of rupture is determined by the microstructures and material properties. A method for modelling the critical temperature difference of rupture of a rectangular specimen is proposed.

1. Introduction

In the last few decades, ceramics have been developed for high-temperature structural materials for aerospace and other structural applications. For example, ultra-high temperature ceramics have attracted diverse attention because of their unique properties of high melting temperature, low density, and good chemical and physical stability at high temperatures. These unique properties make them attractive candidates for use in thermal protection systems and propulsion systems in aerospace applications [1–4]. ZrO_2 ceramics are used to obtain the high fracture toughness of high-temperature structural materials [5,6]. Those ceramics that are used for high temperature applications are usually exposed to severe thermal environments and are often subjected to a rapid heating or cooling process, which can lead to the formation of high thermal stresses. Due to the inherently brittle nature of ceramic materials, those thermal stresses can induce cracking and even lead to failure of the materials. The poor thermal shock resistance of ceramics limits their applications at high temperatures.

The thermal shock of ceramics includes ascending thermal shock and cooling thermal shock. Many studies have been devoted to the understanding and improvement of the thermal shock resistance of ceramics. Most of the studies have investigated cooling thermal shock, as the experiments can be performed simply. A

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commonly used experimental method is water quench testing. At present, the effects of size and geometry of a specimen, cooling medium temperature, water entry posture, surface oxidation, and pre-crack and constraint conditions on the thermal shock resistance of ceramics during cooling have been reported [7–14]. However, compared with cooling thermal shock there are only a few studies about ascending thermal shock owing to the difficulties in performing experiments and high cost. Several common methods include electron beam heating, oxy-acetylene heating and plasma arc heating, which are relatively low cost and simple [15–18]. However, during the ascending thermal shock testing using these methods, the central portion of the specimen is heated rapidly firstly, leading to an uneven temperature distribution across the surface of the specimen, and the target temperature of thermal shock during heating is very difficult to control. This suggests that the error when determining the temperature of the surface of a specimen will be very large. In addition, severe changes in the surface morphology of the shocked specimen accompany the thermal shock, such as surface erosion. Yet, internal cracks in the shocked specimen have not been reported. Some authors have determined the ascending thermal shock resistance of materials based on the degree of changes in the surface morphology of the shocked specimen. However, under this experimental circumstance there are not only just the problems of thermal shock but also the problems of oxidation or erosion. This would affect the obtained results of the effects of some factors on the thermal shock resistance of materials. Some researchers have also used an arc-heated wind tunnel to fulfill the ascending thermal shock testing [19]. Yet, an arc-heated wind tunnel is costly, and its operation is complicated. Currently, the ascending thermal shock resistance of ceramics is a comparatively unexplored scientific field owing to a lack of an efficient method for obtaining thermal shock measurements. The lack of experimental data denies us an essential understanding of ascending thermal shock resistance or even a basic proper experimental evaluation index. Considering that during the causative processes of ceramic materials in high-temperature applications, such as the thermal protection materials in hypersonic vehicles, the failure of the materials usually occurs because of the intense ascending thermal shock, it is therefore essential to conduct research in this important scientific field.

In our previous work [20], we proposed a new, simple and efficient ascending thermal shock testing method. In this method, the specimen is dropped into a thermal environment from a low-temperature environment automatically and momentarily. This solves the existing problems of the commonly used ascending thermal shock testing methods, where the central portion of the specimen is heated rapidly firstly and the target temperature of thermal shock is very difficult to control. Additionally, the surface morphology of the shocked specimen did not change. The method obtained preliminary validation by testing with ZrO_2(3Y) (1 μm, density = 98.6%, Heqishun ceramic Co., Ltd., Chongqing, China) using a specimen size of 4 mm × 12 mm × 68 mm. In this work, taking ZrO_2(3Y) (1 μm, density = 99%, Heqishun ceramic Co., Ltd., Chongqing, China) with different specimen sizes for example (4 mm × 5 mm × 60 mm, 5 mm × 6.5 mm × 60 mm, 6 mm × 7.5 mm × 60 mm and 8 mm × 10 mm × 60 mm), the ascending thermal shock behavior and its characterization are studied systematically by using the previously proposed method; the effects of the target temperature of ascending thermal shock and the specimen size on the thermal shock resistance of materials are studied in detail. The retained flexural strength of the shocked specimen is obtained. SEM images of the microstructures of the fracture surfaces and scans of the internal damage of the shocked specimens are obtained and analyzed. A characterization method for the ascending thermal shock resistance of materials is proposed.

2. Experimental

The ascending thermal shock testing was fulfilled by a new, simple and efficient method. The idea behind the method was to develop a thermal environment firstly, the temperature of which could be controlled easily and accurately. Then, we allowed the specimen to fall momentarily and automatically into this thermal environment from low temperature with the help of a guiding system consisting of a molybdenum wire. This method ensured an accurate target thermal shock temperature and the even distribution of the temperature across the surface of the specimen. The method was performed by refitting a traditional quenching furnace (Fangrui Technology Co., Ltd., Changchun, China) [20].

When doing the ascending thermal shock testing, the heating furnace chamber was heated to the desired temperature and held for 10 min. Afterward, the specimen dropped into the heating furnace chamber and kept there for 2 min. The ascending thermal shock was then fulfilled, and the heating furnace chamber was cooled slowly. In this work, the ZrO_2(3Y) was used as an example in the ascending thermal shock testing. ZrO_2(3Y) (1 μm, density = 99%, Heqishun ceramic Co., Ltd., Chongqing, China) was 3 mol% Y_2O_3 partially stabilized zirconia. The specimens were rectangular bars, the sizes of which were 4 mm × 5 mm × 60 mm, 5 mm × 6.5 mm × 60 mm, 6 mm × 7.5 mm × 60 mm and 8 mm × 10 mm × 60 mm. Due to a limitation of the used equipment, the highest ascending thermal shock temperature was 1600 °C. The effects of the specimen size on the ascending thermal shock behavior were investigated firstly by observing the visible changes of the shocked specimen with above mentioned four kinds of sizes after being thermally shocked at 1600 °C. Afterward the systematic and rational experimental thermal shock temperature points were used to determine the critical temperature differences of rupture of specimens with different sizes and show the size effect in more detail. The retained flexural strength of the shocked specimen was tested at room temperature by a three-point bending test using a 50 mm span and a cross-speed of 0.5 mm min^{-1} (WDW-100, China). A minimum number of four specimens were tested for each temperature point, and the average value was calculated. The SEM images were used to analyze the microstructures of the fracture surfaces of the shocked specimens. The scans of the internal damage of the shocked specimens were obtained by using an ultrasonic flaw detector.

3. Results and discussion

As observed from Fig. 1, visible changes are not found on the surface of the shocked specimen with a size of 4 mm × 5 mm × 60 mm after being thermally shocked at 1600 °C. However, the fracture of the specimens with sizes of 5 mm × 6.5 mm × 60 mm, 6 mm × 7.5 mm × 60 mm and 8 mm × 10 mm × 60 mm occur during the ascending thermal shock. The specimen with a size of 5 mm × 6.5 mm × 60 mm fractures into two parts, and the one with a size of 6 mm × 7.5 mm × 60 mm fractures into some pieces, while the other one with a size of 8 mm × 10 mm × 60 mm fractures into fragments. However, there are no visible cracks on the surfaces of fractured specimens. As is well known, during ascending thermal shock, the interior of the specimen suffers from tensile stress, while the surface of the specimen suffers from compressive stress. Thus, the cracking should occur in the interior of the specimen firstly.

From Fig. 2, we can observe the cracks in the fracture surfaces of the tested specimens. It should be noted that although the shocked specimen with a size of 4 mm × 5 mm × 60 mm does not fracture during the ascending thermal shock, cracks formed due to thermal shock can be found on fracture surface. For the shocked specimens