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Short communication

High-temperature flexural creep of ZrB_2–SiC ceramics in argon atmosphere

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Abstract

Four-point flexure creep deformation of ZrB_2–30 vol% SiC ceramics in argon atmosphere under a static load of 19 MPa for 0–100 h at 1500 and 1600 °C was investigated. The strain rate at 1600 °C was 3.7 times higher than that at 1500 °C. Microstructural evolution during creep consisted of nucleation and growth of triple-point cavitations which were always associated with SiC particles. Due to the low stress, only isolated cavitations were nucleated, and no microcracks were formed. For up to 100 h at 1500 and 1600 °C, the grains maintained their size and shape. The cavitations in both size and number showed no obvious difference from 26 to 100 h at 1500 °C, whereas that showed a significant increase from 26 to 100 h at 1600 °C. Present study suggested that ZrB_2–30 vol% SiC exhibited relatively good microstructural stability and creep resistance at 1500 °C in argon atmosphere.

Keywords: C. Creep; ZrB_2–SiC; Four-point flexure; Microstructure; Cavitations

1. Introduction

During the past decade, the ZrB_2–SiC ceramics have been investigated extensively, and most of works have been conducted on densification, mechanical, and oxidation behavior [1–6]. Only a limited experimental work involved creep behavior of ZrB_2–SiC ceramics. Talmy et al. studied the flexure creep deformation of ZrB_2–SiC ceramics containing 0–50 vol% SiC for very short test period of up to 5 h in oxidizing atmosphere as a function of temperature (1200–1500 °C), stress (30–180 MPa), and SiC particle size (2 and 10 μm) [7]. Results showed the creep rate increased with increasing SiC content, temperature, and stress, but with decreasing SiC particle size, especially at temperatures above 1300 °C. Also, Han et al. studied high temperature deformation of ZrB_2–SiC–AlN ceramic composites, revealing that the composites had excellent high temperature deformation property at 1900 °C [8].

Most of studies on creep behavior of non-oxide ceramics (SiC, Si_3N_4, ZrB_2–SiC) were carried out in oxidizing atmosphere [7,9,10]. During creep, the non-oxide ceramics would readily oxidize and could be substantially enhanced due to the applied stress. For example, a continuous oxide layer of $Y_2Si_2O_7$ formed at the surface of the liquid-phase-sintered SiC ceramics containing AlN–Y_2O_3 additives during elevated temperature creep testing in air [9]. In the ZrB_2–50 vol% SiC ceramics after creep testing at 1300 °C and 100 MPa for 4 h, a 50 μm oxidation layer was formed on the compressive side, and 80 μm on the tensile side [7]. Therefore, virtually the obtained creep behavior was a combination of non-oxide ceramics and oxide layer. Thus, the true creep response and controlling mechanisms of non-oxide ceramics could not be obtained in oxidizing atmosphere.

The present work investigated the four-point flexure creep deformation of ZrB_2–30 vol% SiC ceramics in argon atmosphere under low static load (19 MPa) for up to 100 h at 1500 and 1600 °C. To shed some light into the creep controlling mechanisms without the introduction of oxidation factor, the microstructure evolution of the sample before and after creep was studied in detail via scanning electron microscopy.

2. Experimental procedure

The raw materials used in this study were self-synthesized ZrB_2 powders ($D_{50} = 0.6$ μm, oxygen content was about 0.7 wt%), and commercial α-SiC powders ($D_{50} = 0.45$ μm, purity 98.5%, Changle Xinyuan Carborundum Micropowder

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Co. Ltd., Changle, China). The ZrB_2 powders were synthesized by our group. The details about processing and characteristics of ZrB_2 powders could be found in Ref. [11]. The SiC powders were added to the synthesized ZrB_2 powders to fabricate ZrB_2–30 vol% SiC ceramics. The starting mixture was mixed for 24 h in a polyethylene jar using ethyl alcohol and Si_3N_4 balls, and dried by rotary evaporation. After being dried, the mixed powder was ground to −200 mesh and then placed in a graphite die with a BN coating. The powder compact was hot pressed at 2000 °C for 60 min under a pressure of 30 MPa in an argon atmosphere with a heating rate of 10 °C/min.

High-temperature deformation was conducted in four-point flexure with outer span of 30 mm and inner span of 10 mm under an applied static load (19 MPa) provided by the gravitation of a tungsten block. The sample with dimensions of 3 mm × 4 mm × 35 mm was heated at a rate of 50 °C/min to the test temperature (1500 and 1600 °C) and held for up to 100 h in the graphite element furnace in an argon atmosphere. The deformation displacement of sample was measured by absolute digimatic indicator (Mitutoyo, Japan). It should be noted that the creep displacement was measured at room temperature. The creep specimens were cooled down to room temperature under the applied load. Based on the creep displacement, the strain (ε) in four-point bending creep test was calculated using the following equation [12]:

$$\varepsilon = \frac{4h}{s^2}\delta \qquad (1)$$

where δ is the displacement of the center relative to the inner rollers, h is the thickness of the sample, and s is inner roller span. The microstructure of the samples before and after creep deformation was studied by scanning electron microscopy (SEM, Hitachi S4800).

3. Results and discussion

Fig. 1 shows the creep curves obtained at 1500 and 1600 °C under a load of 19 MPa for 100 h. The two curves showed the primary and secondary stages of creep. The tertiary stage of creep was not observed presumably due to the low applied stress and/or short test time. The second stage where the strain rate was constant was reached after a 16 h period for 1500 and 1600 °C. In the second stage, the strain rates were estimated to be approximately 1.9×10^{-9} and $7.1 \times 10^{-9}\ s^{-1}$ for 1500 and 1600 °C, respectively. This meant that the strain rate at 1600 °C was 3.7 times higher than that at 1500 °C.

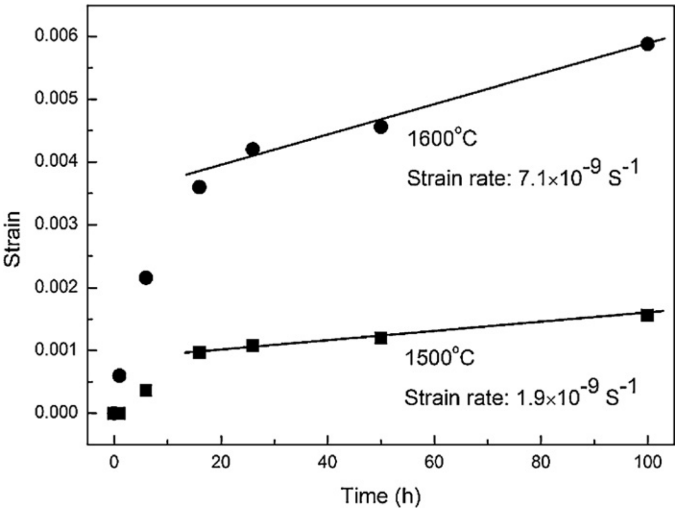

Fig. 1. Strain in ZrB_2–30 vol% SiC ceramics during creep at 1500 and 1600 °C under the stress of 19 MPa.

Due to the limitations of the present bending apparatus, the creep curves were not recorded continuously. As a result, study of the creep mechanisms via the stress exponent approach was not able to be achieved. However, it was surmised that the microstructural observations might provide an insight into the creep controlling mechanisms. Fig. 2 shows the microstructures of as-sintered and heat-treated samples at 1600 °C for 1 h under an argon atmosphere in the absence of applied load. The pores were absent in the as-sintered sample, indicative of fully dense of hot-pressed ZrB_2–30 vol% SiC ceramics. After 1 h of heat treatment without load in argon, clear grain boundaries appeared due to etching process and no pores were observed. This suggested that no volatile species existed in the as-sintered ZrB_2–30 vol% SiC ceramics.

Fig. 3 shows the microstructures of the tensile side of crept sample at 1600 °C for 26 h under static load of 19 MPa in an argon atmosphere. In the presence of applied load, some isolated

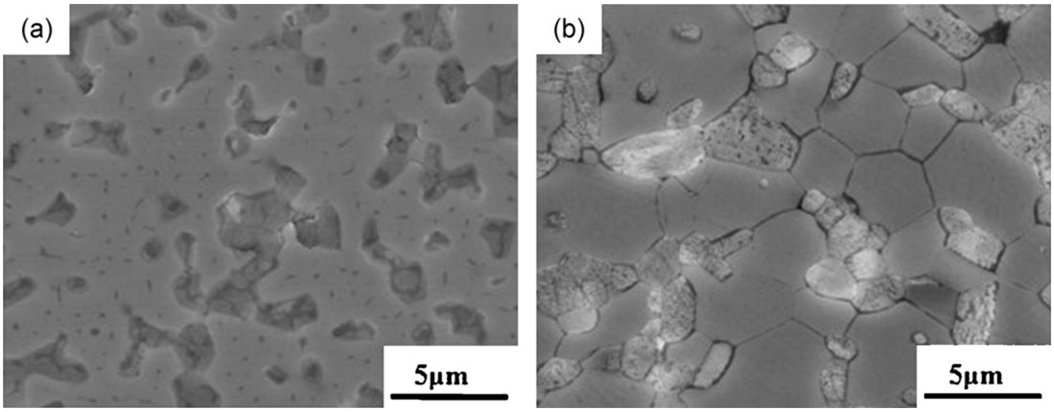

Fig. 2. Microstructures of as-sintered sample (a) and heat-treated sample (b) at 1600 °C for 1 h under an argon atmosphere in the absence of applied load.

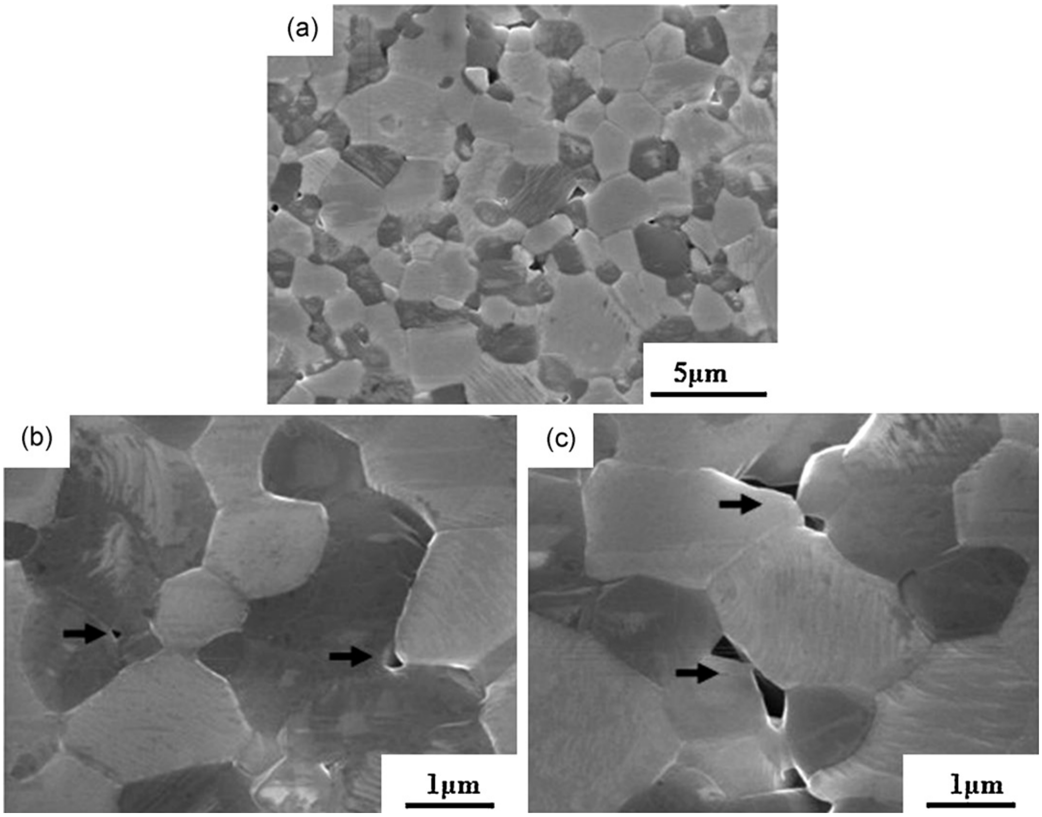

Fig. 3. Microstructures of the tensile side of crept sample at 1600 °C for 26 h under static load of 19 MPa in an argon atmosphere: (a) low magnification; (b and c) high magnification.

cavitations were observed at the multigrain junctions (indicated by arrows in Fig. 3(b) and (c)). There was also evidence for the nucleation and growth of cavitations. Similarly, triple-point cavitations were also observed in the many other ceramics systems during creep [13–15]. Such triple-point cavitations could form due to grain-boundary sliding. More studies are required to determine the creep characteristics and mechanisms of the ZrB_2–30 vol% SiC ceramics under an argon atmosphere.

Furthermore, it was found that cavitations were mostly located at the triple points of SiC grains or the triple points of SiC grains and ZrB_2 grains, as seen in Fig. 3. Observations suggested that the cavitations were always associated with SiC grains. In the creep of zirconia–20 wt% alumina composite, Owen et al. found that triple-point cavitations were associated frequently with alumina grains [15]. It was concluded that the alumina particles acted as the hard phase in the zirconia–alumina composite, so that cavities were nucleated in association with hard alumina particles and grain boundary sliding. In the creep of alumina–17 vol% silicon carbide composite, cavities were associated with SiC particles which acted as a hard phase [16]. Therefore, in the creep of ZrB_2–30 vol% SiC ceramics, the cavitations in association with SiC particles might be also attributed to the fact that SiC particles acted as the hard phase in the composite.

In the present work, the angular cavitations were only observed at the triple junctions. However, cracks were observed on the tensile side of ZrB_2–25 vol% SiC after creep at 1400 °C and 100 MPa for 5 h [7]. The low applied stress (about 19 MPa) employed in the present study might only cause the nucleation of isolated and angular cavitations, whereas the high stress (about 100 MPa) could lead to the nucleation and growth of microcracks reported. Similar trend was also observed by Wilkinson et al. for the four-point flexure creep of hot-pressed alumina [17]. At high stress ($\geq$100 MPa), many microcracks were nucleated in alumina, whereas many angular cavitations were nucleated at the triple grain junctions at the lowest stress levels ($\leq$40 MPa).

Fig. 4 shows the microstructures of the tensile side of crept sample at 1500 and 1600 °C under static load of 19 MPa in an argon atmosphere at 26 and 100 h, and the cavitations were indicated by arrows. Even though creep temperature increased from 1500 to1600 °C and creep time increased from 26 to 100 h, no apparent grain growth took place. The grains maintained their mean size and their shape, which suggested the good microstructure stability. With increase of the creep time from 26 h to 100 h at1500 °C, the characteristics of cavities in both size and number basically showed no change. When the creep temperature increased from 1500 °C to 1600 °C, the cavitation features of crept sample changed. Unlike the crept sample at 1500 °C, both size and number of cavitations showed a significant increase with increase of the creep time from 26 h to 100 h at 1600 °C. Nonetheless, even at 1600 °C there were no formation of cracks due to the growth and coalescence of creep cavities under the stress and test time employed. Creep results of present study suggested that the ZrB_2–30 vol% SiC ceramics exhibited relatively good microstructural stability and creep resistance at 1500 °C in argon atmosphere. However, more detailed creep studies are needed in order to shed the light of

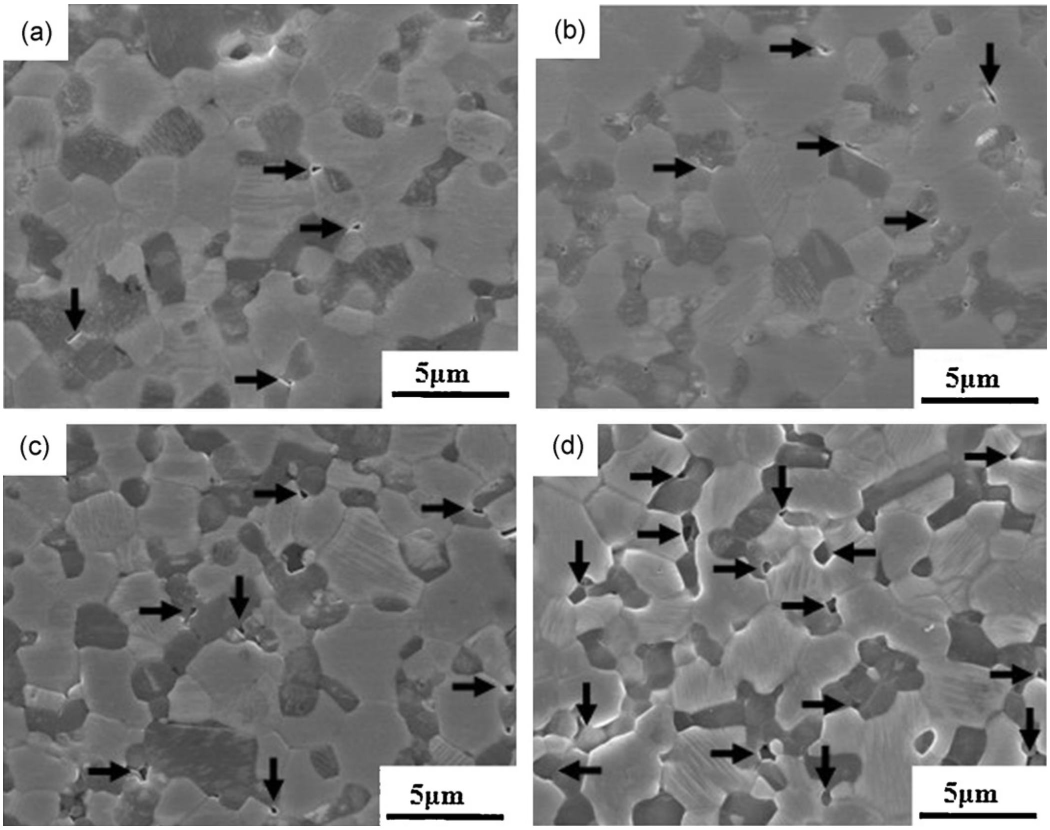

Fig. 4. Microstructures of the tensile side of crept samples at 1500 ((a) 26 h; (b) 100 h) and 1600 °C ((c) 26 h; (d) 100 h) under static load of 19 MPa in an argon atmosphere.

creep controlling mechanisms and life limiting factors of ZrB_2–30 vol% SiC ceramics under the controlled environment.

4. Conclusion

The high-temperature four-point flexure creep deformation of ZrB_2–30 vol% SiC ceramics in argon atmosphere under a static load of 19 MPa for 0–100 h at 1500 and 1600 °C was investigated. The steady-state creep rate at 1600 °C was 3.7 times higher than that at 1500 °C. The microstructural evolution during creep consisted mainly of nucleation and growth of angular cavitations at the triple junctions. These cavitations were always associated with SiC particles. Due to the low stress, only isolated cavitations were nucleated, and no microcracks were formed. For up to 100 h of test time at 1500 and 1600 °C, the grains maintained their size and shape. The cavitations in both size and number showed no obvious difference from 26 to 100 h at 1500 °C, whereas that showed a significant increase from 26 to 100 h at 1600 °C. Present study suggested that ZrB_2–30 vol% SiC exhibited relatively good microstructural stability and creep resistance at 1500 °C in argon atmosphere.

Acknowledgments

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