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High temperature ductility and cavitation behaviour of hot isostatically pressed (HIP) ZrO_2/Al_2O_3 composite containing 40 wt.% Al_2O_3

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Abstract

In this study, experiments were conducted to investigate the effect of hot isostatic pressing (HIP) on superplastic ductility of ZrO_2/Al_2O_3 composite containing 40 wt.% Al_2O_3 at a constant strain rate of 1×10^{-4} s^{-1} and 1723 K. The results show that superplastic ductility obtained from HIPed specimen is higher than that of sintered one. The reason for this ductility enhancement is explained in terms of elimination of agglomerates in the sintered specimen. Also the effect of hot isostatic pressing on cavitation behaviour is quantitatively examined. It is shown that extensive internal cavities form during superplastic flow in sintered and HIPed specimens. The amount and size of cavities decrease with hot isostatic pressing stress.

Keywords: A. Hot isostatic pressing; B. Composites; D. ZrO_2; D. Al_2O_3; Cavitation; Superplastic ductility

1. Introduction

In the 1950s extensive efforts were made on hot fabricating ceramics using conventional metallurgical processes such as extrusion, rolling and forging. The aim was to produce near-net-shape parts in order to avoid the expensive machining of ceramics. The behaviour of a number of structural oxides, including CaO, MgO, SiO_2, ZrO_2, BeO, ThO_2 and Al_2O_3 was studied [1]. As a result of these works, an improved understanding of ceramic deformation was developed but certain problems, and in particular the requirement for relatively high forming temperatures, still existed. For example, the temperatures required for hot forging Al_2O_3 was found to be about 1900 °C which is extremely high from a practical point of view [1]. Subsequently, the concept of thermomechanical processing of ceramics was more or less abandoned.

Recently, two major technical advancements have changed this situation. Firstly, ceramic powder technology has been greatly advanced and the quality of ceramic powders has greatly improved. Ceramics of fine (less than 1 μm grain size) and more consistent microstructures are routinely prepared. As a result, fine-grained ceramic powders present the possibility for deformation to be conducted at decreased temperatures and increased deformation rates. Secondly, fine-grained superplasticity in metallic alloys has been extensively studied and has found commercial application [2]. As a result of these two advances, the concept of hot forming and in particular superplastic forming of ceramics has become an area of intense study. Current understanding of ceramic superplasticity has advanced to the stage where superplastic forming/stretching of ceramics may now be considered to be a viable manufacturing technology and has been envisioned "as a future technology in to the next century".

Superplasticity has now been demonstrated for many ceramics and ceramic composites, including yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) [3], yttria-doped Al_2O_3 [4], hydroxyapatite [5], β spodumene glass ceramics [6], Al_2O_3-reinforced YTZP [7,8], SiC-reinforced Si_3N_4 [9] and iron-iron carbide ($Fe\text{-}Fe_3C$) [10] composites. Most of the above materials have involved tension, compression or in rare cases punch-forming tests.

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Several commercial processes in metal and ceramic industries have taken advantage of the high deformability of the superplastic state to form intricate, large-scale components directly into net shapes [11]. To encourage the commercial applicability of superplastic forming of ceramics, it is desirable that sufficient superplastic elongation can be attained at high strain rates and at relatively low temperatures. A large amount of work has been carried out to assess the effect of test temperature, strain rate, grain size, prestraining or doping with transition metal oxides on superplastic behaviour in a range of ceramics [11–13]. The present study was conducted with the aim of investigating the effect of hot isostatic pressing (HIP) on superplastic ductility of ZrO_2/Al_2O_3 composite containing 40 wt.% Al_2O_3 at a constant strain rate of 1×10^{-4} s^{-1} and 1723 K.

2. Experimental procedures

The materials investigated in the present work are 3 mol% yttria-stabilized tetragonal zirconia (ZrO_2) powder and a high purity (>99.999%) α-Al_2O_3 powder, supplied by Mandoval Ltd. Zirconia Sales (U.K.) Ltd. The average particle sizes are 0.3 μm for ZrO_2 and 0.4 μm for α-Al_2O_3. The chemical compositions are listed in Table 1.

ZrO_2/Al_2O_3 composite containing 40 wt.% Al_2O_3 were prepared by dispersing the designated amount of alumina powder and zirconia powder in distilled water containing a dispersing agent (Dispex A40, supplied by Allied Colloids UK). This mixture was ball milled for 10 hours to break-up agglomerates in a plastic container using zirconia balls. The mixed composite powders were then dried and ground prior to consolidation.

The specimens for superplastic tests were uniaxially pressed to net-shape at 40 MPa and then isostatically cold-pressed at 300 MPa. The green compacts were sintered to 98.4% of theoretical density at 1750 K by pressureless sintering (sintered specimen) or pressureless pre-sintering at 1750 K followed by hot isostatic pressing (HIPed specimen). Hot isostatic pressing was performed in Ar gas with an ABB HIP system at 1723 K and 150 MPa for 30 min. The density of the HIPed specimen was 99.4% of the theoretical density. The densities of both sintered and hot isostatically pressed specimens were calculated from the volume and weight. The details of specimen preparation procedures are illustrated in Fig. 1.

High temperature uniaxial tensile tests were carried out in air using an Instron 4505 testing machine. A single zone vertical split furnace (supplied by Carbolite Furnaces Ltd.) with molybdenum disilicide elements was mounted on the crosshead of the test frame; tensile load was applied using high density sintered alumina rods in a pin loading mechanism. Careful specimen alignment was essential to avoid fracture on loading. After achieving the desired (uniform) test temperature, usually at a heating rate of 423 K/h, the assembly was held at that temperature for about 10 min. A small tensile load was then applied on the specimen as a pre-load and the alignment was checked before testing. Deformation was continuously monitored using a computerized system equipped with a data acquisition facility that allowed tests to be controlled under a constant strain rate. The work reported here involves a test temperature of 1723 K and a strain rate of 1×10^{-4} s^{-1}.

Microstructural observations were carried out using a Philips scanning electron microscope (SEM 525) for grain size measurements. For cavitiy characterization, specimens were mechanically polished to 1 μm diamond finish and quantitatively examined using an optical microscope connected to an image analysis system (Magiscan) with software capable of counting and sizing discrete cavities by automatically scanning any selected region of the image. The region scanned was set as a rectangular grid with an area of 3.6×10^{-2} mm^2 . Measurements were taken at mid-section in a region between 2.50 and 3.00 mm from the center of the gauge section using 8 measurements on both sides of the center location (16 grid measurements in total per specimen). To avoid problems in the cavity count, including minor artefacts introduced during specimen preparation, a limit of resolution was set so that no cavities were counted having areas less than 1 μm.

3. Results and discussion

To investigate the effect of HIPing on superplastic ductility, sintered and HIPed specimens were tested at 1×10^{-4} s^{-1} and 1723 K. True stress–true strain relationships for a sintered specimen and a HIPed specimen are shown in Fig. 2. It is seen that the strain hardening

Table 1
Chemical composition of the powders

Materials	Composition in wt.%							
	ZrO_2 (+HfO_2)	Y_2O_3	Al_2O_3	SiO_2	TiO_2	Fe_2O_3	Na_2O	CaO
t-ZrO_2	93.8	5.4	0.25	0.11	0.12	0.003	0.02	0.06
α-Al_2O_3	–	–	99.9	0.04–0.08	–	0.01–0.02	0.08	–

Mixing of ZrO_2 and Al_2O_3 powders in distilled water

⇓

Ball milling for 10 hours

⇓

Drying at 373 K and grinding

⇓

Uniaxiall pressing at 40 MPa

⇓

Cold isostatic pressing at 300 MPa

↙ ↘

Pressureless sintering at 1750 K for 1 hour | Presintering at 1750 K for 30 minutes

⇓

Hot isostatic pressing at 150 MPa and 1723 K

Fig. 1. Flow chart showing the procedures in specimen preparation.

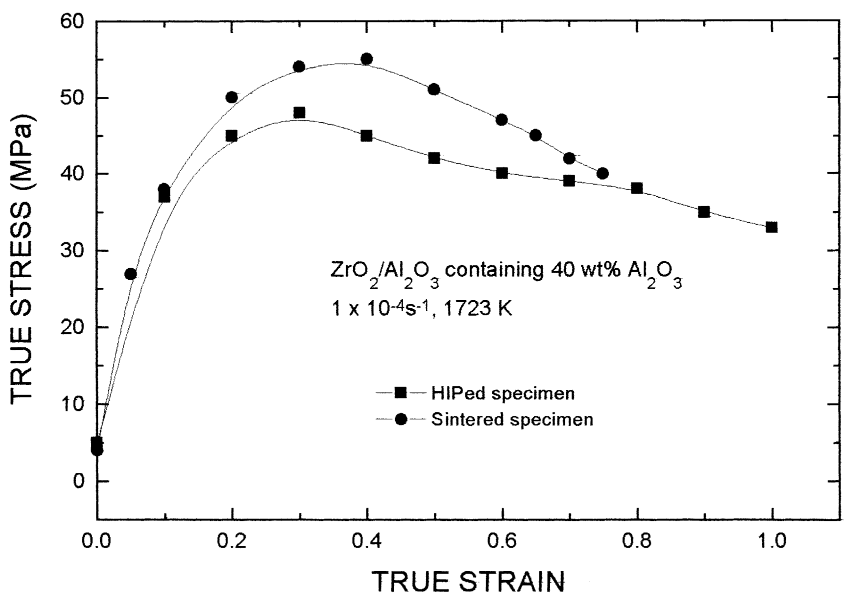

Fig. 2. True stress–true strain relationships for sintered and HIPed specimens of ZrO_2/Al_2O_3 composite containing 40 wt.% Al_2O_3 tested at 1×10^{-4} s^{-1} and 1723 K.

of the sintered specimen is greater than that for the HIPed specimen, although the grain growth in the former is less than that for the latter. This different strain hardening may be due to the different modes of cavitation in the two specimens. It is also seen that significant enhancements in elongation and slight decreases in flow stress were obtained by HIPing specimen. Notably, an elongation to failure of 145% was achieved from the HIPed specimen, compared to 110% in the sintered specimen. Fig. 3 shows the profiles of sintered and HIPed specimens deformed at 1×10^{-4} s^{-1} and 1723 K. For comparison, an untested specimen was also included. The higher ductility is clearly visible in specimen C and there was no evidence of necking within the gauge length of the deformed specimens.

Enhancement of superplastic ductility in ceramics has been considered to be due to a number of contributing factors, including the presence of a low viscosity grain boundary (glassy) phase or doping with transition metal oxides. These additives possibly act in a multiple role as sintering aids, grain growth inhibitors and modifiers of grain boundary strength and grain boundary chemistry. Conventionally, reducing the strain rate or increasing the test temperature (but necessarily avoiding grain growth) also promotes larger elongations at lower stress levels. In the present work, it is shown that HIPing improves superplastic ductility. The processing defects and agglomerates are responsible for the decrease in superplastic ductility of the sintered specimen. Improvement of superplastic ductility in the HIPed

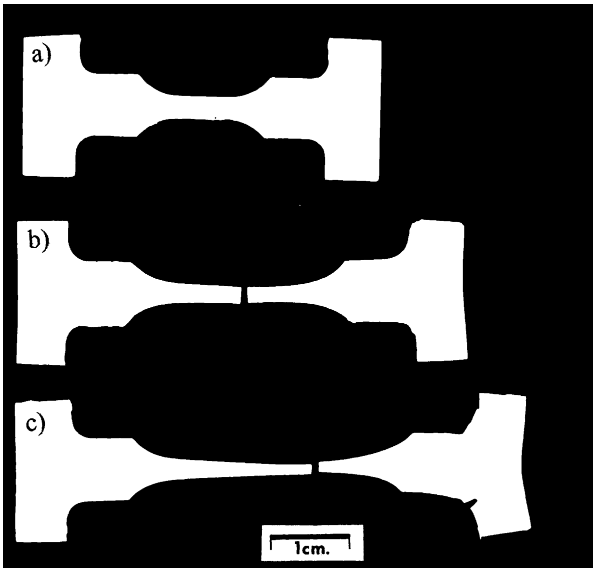

Fig. 3. Specimen profiles of ZrO_2/Al_2O_3 composite containing 40 wt.% Al_2O_3 deformed at 1×10^{-4} s^{-1} and 1723 K (a) undeformed specimen (b) sintered specimen, $e_f = 110\%$ (c) HIPed specimen, $e_f = 145\%$.

specimen can be explained by elimination of defects and agglomerates introduced during powder processing and shaping processes. Large agglomerates remained in ZrO_2/Al_2O_3 composite containing 40 wt.% Al_2O_3 after inefficient ball milling and dry powder consolidation. Lange et al. [14] demonstrated that dry powder routes to powder consolidation can produce large agglomerates which result in large crack-like voids due to different sintering of matrix and agglomerates. In dry powder routes, agglomerates are apparently formed either by the spontaneous attraction of particles due to Vander Waals forces or by particle cohesion due to surface tension effects produced by adsorbed atmospheric water. Dry powder consolidation also yields less efficient packing of the powder and this feature is known to retard effective sintering.

In the sintered specimen, agglomerates shrank away from the surrounding powder matrix during sintering causing crack-like voids responsible for early fracture and leaving big pores in the microstructure after sintering. The results of microstructural observations showed that hot isostatic pressing mostly eliminates processing defects and agglomerates in the presintered specimen (Fig. 4). The present author [15] showed that the elimination of agglomerates can also be achieved by slip casting in which the powders are homogeneously dispersed in a slurry giving efficient packing of powders.

To determine whether cavity morphology is affected by hot isostatic pressing, sintered and HIPed specimens were pulled to predetermined strain of 110% at 1×10^{-4} s^{-1} and 1723 K. Fig. 5 shows distribution of cavities in the sintered and the HIPed specimens. Extensive internal cavitation developed in both specimens. As the extent of cavitation was different along the gauge length, the photomicrographs in Fig. 5 were taken at the mid-point of the gauge length. It has been reported by Okada et al. [16] and Wadsworth et al. [17] that, in ZrO_2/Al_2O_3 composite, there are three different types of grain (or particle) boundaries i.e. ZrO_2–ZrO_2, ZrO_2–Al_2O_3 and Al_2O_3–Al_2O_3 grain boundaries. Due to the differences in chemical compositions and orientations, the sliding mobility for each boundary combination is expected to be different. In the fine-grained superplastic materials, the deformation processes involves not only grain boundary sliding but also grain rotation to accommodate large elongations. Different sliding mobilities at ZrO_2–ZrO_2, ZrO_2–Al_2O_3, and Al_2O_3–Al_2O_3 interfaces are expected to result in strain incompatibilities and thus cavitation. In addition, grain growth of alumina occurred at a faster rate than that of zirconia in ZrO_2/Al_2O_3 composite containing 40 wt.% Al_2O_3 causing strain incompatibilities and higher sliding mobilitiy at zirconia and alumina interfaces.

There are two conclusions that can be made from Fig. 5. First, the amount and size of cavities in the HIPed specimen are lower than that of the sintered specimen; this contributes to an increase in ductility. Secondly, in the sintered specimen, cavity interlinkage transverse to the tensile axis occurs occasionally; whereas in the HIPed specimen, the cavities are almost exclusively elongated parallel to the tensile axis; some of these cavities have become cavity stringers. These differences are quantitatively presented in Fig. 6, that shows the number of cavities as a function of cavity area. Hot isostatic pressing considerably reduced the number of cavities and cavity size compared to the sintered specimen. The reason for the decrease of cavitation in the HIPed specimen is that cavity initiation is delayed as well as nucleation due to the absence of defects and agglomerates which are favourably sites for cavity nucleation. Agglomeration had occurred in the sintered specimen and increased cavity nucleation was associated with this agglomeration. Conversely, in the case of hot isostatic pressing mostly eliminated agglomeration cavitation is reduced leading to increased elongation.

4. Summary

The effect of hot isostatic pressing (HIP) on superplastic ductility of ZrO_2/Al_2O_3 composite containing 40 wt.% Al_2O_3 at a constant strain rate of 1×10^{-4} s^{-1} and 1723 K was investigated. It is shown that superplastic ductility obtained from HIPed specimen was higher than that of sintered specimen. The elongation to failure was 145% in the HIPed specimen, whereas in the sintered specimen, elongation to failure of 110% was obtained. The increases in ductility in the HIPed specimen can be attributed to the elimination of agglomerates introduced during powder processing and shaping

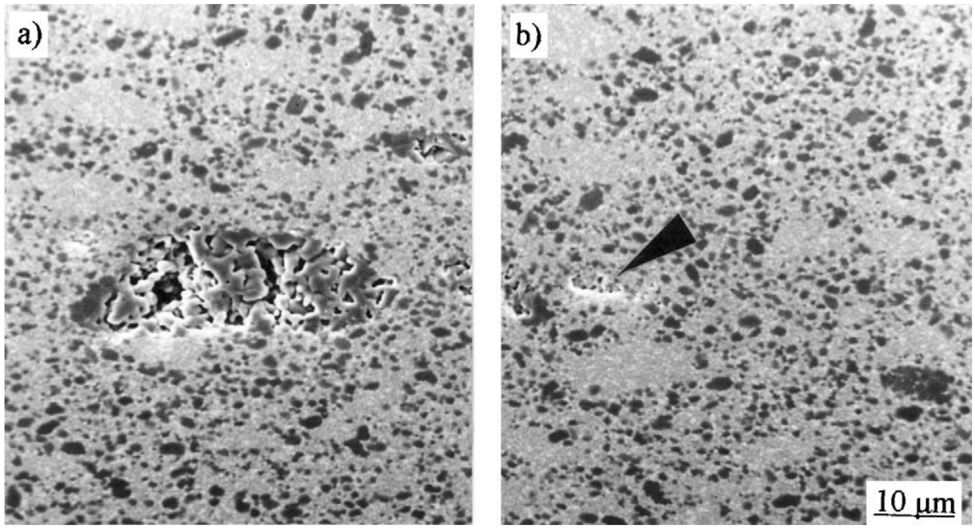

Fig. 4. Agglomeration in (a) the sintered specimen (b) the HIPed specimen. Micrographs in two pictures were taken from the same location.

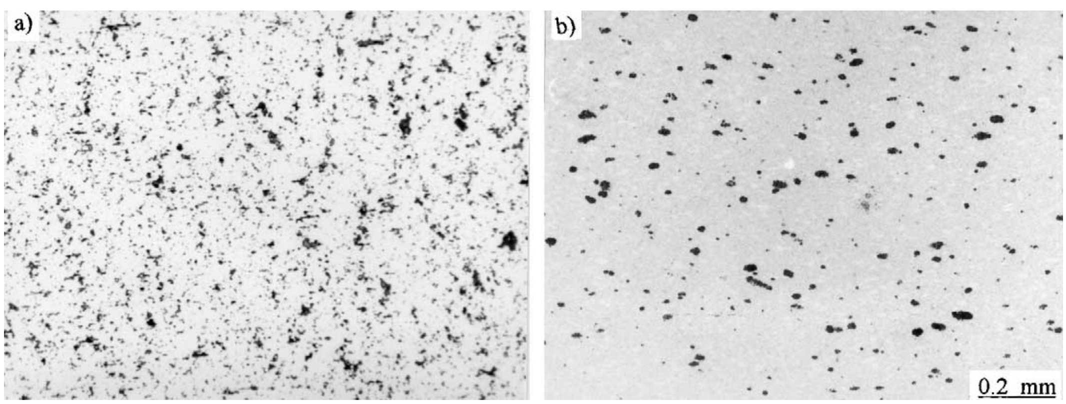

Fig. 5. Appearance of internal cavities in (a) the sintered specimen (b) the HIPed specimen; the tensile axis is horizontal.

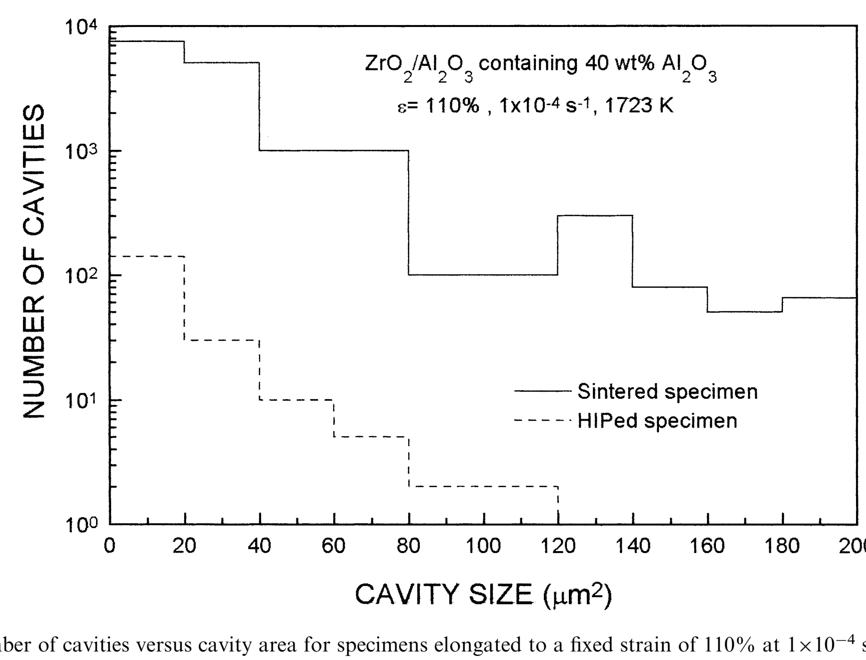

Fig. 6. Number of cavities versus cavity area for specimens elongated to a fixed strain of 110% at 1×10^{-4} s^{-1} and 1723 K.

processes. Also the effect of hot isostatic pressing on cavitation behaviour was quantitatively examined. It is shown that extensive internal cavitation developed during superplastic flow in the sintered and the HIPed specimens. The amount and size of cavities is decreased by hot isostatic pressing. The cause of the decrease of cavitation in the HIPed specimen is that cavity initiation and nucleation are prevented due to the absence of defects and agglomerates which are favourably sites for cavity nucleation.

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