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# A quantitative assessment of cavities in 3 mol% yttria-stabilized tetragonal zirconia specimens containing various grain size

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**Abstract**

It is well established that cavitation occurs in most materials during superplastic deformation, and cavitation has an effect on elongation to failure and mechanical properties of the materials after deformation. The cavities most likely nucleate at grain boundary particles or at triple points in quasi-single phase materials, at triple points and grain boundary ledges in microduplex materials and at particulate or whisker reinforcement in metal matrix composites. To minimize cavitation during superplastic flow it is necessary to exercise sound microstructural control. The starting materials should be processed to develop a fine uniform grain size and if dispersions are present, these should be fine and uniformly dispersed. In the present study, the effect of grain size on superplastic deformation and cavity formation in 3 mol% yttria-stabilized zirconia polycrystal has been examined. Also the distribution of cavity size and circularity have been quantitatively determined as a function of grain size. The results demonstrated that extensive internal cavities formed during high temperature deformation and the amount of cavitation increased with increasing grain size. 

*Keywords:* B. Grain size; Superplastic deformation; Cavitation; 3Y-TZP

## 1. Introduction

Tetragonal zirconia polycrystals containing 2–4 mol% yttria (Y-TZP) have been reported to be superplastic at temperatures above 1573 K [1]. For more than a decade, it has been known that Y-TZP can be produced with a fine-grained microstructure. The need for achieving a submicrometer grain size was initially motivated by the pursuit of transformation toughening [2]. Ultrafine grains are required to avoid spontaneous tetragonal to monolinic transformation, for which nucleation is grain-size dependent [3]. The subsequent use of this material as a tough and strong ceramic has stimulated the industry to provide powders of excellent sinterability. These commercially available powders can be readily processed to obtain dense ceramics with a grain size ranging from 0.2 to 0.5 μm, sufficiently fine to allow superplastic deformation at a temperature above 1573 K. It is for this reason that Y-TZP has become almost the universal choice for demonstrating ceramic superplasticity in the last few years.

Cavitation during superplastic deformation is an important practical concern, not only because it affects the optimum elongation but also because of the deterioration in the subsequent room-temperature properties. Experimental results indicate that even small levels of cavitation may some times lead to a dramatic degradation in mechanical properties [4]. Most ceramics cavitate during superplastic deformation and cavitation proceeds generally by three stages, namely nucleation, growth and coalescence of voids under the effect of flow stress. Cavities either nucleate or develop from pre-existing defects and their growth, coalescence and interlinkage leads to premature failure. The factors influencing cavity nucleation during superplastic flow include those relating to microstructure such as grain boundary particles, grain size, phases and those associated with deformation conditions, such as strain, strain rate, stress and temperature. Four types of cavity morphology have been observed; these are spherical voids, cavities elongated parallel to the tensile stress, those that coalesce at locations perpendicular to the tensile stress and crack-like cavities that propagate normal to the tensile stress.

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Large superplastic elongations are achieved when cavities elongate parallel to the tensile direction of loading [5]. In 3Y-TZP, there have been numerous investigation of various factors, such as strain rate, strain, test temperature and prestraining, which affect cavity formation in a range of ceramics [6–10].

In the present study, the effect of grain size on superplastic deformation and cavity formation in 3 mol% yttria-stabilized zirconia polycrystal has been examined. Also the distribution of cavity size and circularity have been quantitatively determined as a function of grain size.

## 2. Experimental: materials and procedures

The material used was fine-grained 3 mol% yttria-stabilized tetragonal zirconia (3Y-TZP) supplied by Mandoval Ltd. Zirconia Sales (UK) Ltd. The chemical composition of the powder was 5.4 wt.% $Y_2O_3$ (equivalent to 3 mol% $Y_2O_3$ ), 93.8 wt% $ZrO_2$ and the following impurities (wt.%): $Al_2O_3$ 0.25, $SiO_2$ 0.11, $TiO_2$ 0.12, $Fe_2O_3$ 0.003, $Na_2O$ 0.02 and CaO 0.06. The powder had an initial grain size of 0.2 μm.

A slip casting method was used for the preparation of tensile specimens. This method allowed the economical production of complex, net shapes that required no machining. The slip casting slurry was prepared by dispersing the powder (3Y-TZP) in distilled water with a dispersing agent (Dispex A40); the slurry was then wet ball milled for 4 h to obtain a good dispersion. The milled slurry was injected by a syringe into a plaster mould. Cast specimens were released from the mould after ~10 min and then air dried at ~25 °C for a few days. These specimens were presintered at 950 °C to make them more handleable and smooth surfaces were obtained by carefully grinding off any casting protrusions. Specimens were then pressureless sintered in air at 1250 °C to achieve tensile specimens with ~99% of theoretical density. Density measurement were made by the Archimedes displacement method.

High temperature, uniaxial, tensile tests were carried out in air using an Instron 4505 testing machine. A single zone vertical split furnace 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 150 °C/h, the specimen was held at that temperature for ~10 min before loading. The small tensile load was then applied on the specimen as a pre-load and the alignment 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. Specimens tested were either pulled to failure without interruption or elongated to a predetermined strain. The work reported here involves a test temperature of 1400 °C and strain rate of $1\times10^{-4}$ $s^{-1}$.

Microstructural observations were carried out using a Philips scanning electron microscope (SEM 525) for grain size measurements. For cavity characterization, specimens were mechanically polished to 1 μm diamond finish and the surfaces quantitatively examined using 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 $3.6\times10^{-2}$ $mm^2$ . Measurements were taken at mid-section in a region between 2.50 and 3.00 mm from the center of the gauge section using eight measurements either side 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. Experimental results and discussion

Effect of grain size on superplastic flow To determine the effect of initial grain size on superplastic flow in 3Y-TZP, specimens were thermally annealed after sintering at various temperatures and times to produce various initial grain size. Table 1 shows the annealing temperatures and times employed to obtain various grain sizes. Microstructures of thermally etched sections of annealed samples having an initial grain size of: (1) 1.51 μm, (2) 0.82 μm and (3) 0.47 μm are shown in Fig. 1. Substantial increases in grain size were seen at higher annealing temperatures and longer annealing times.

Table 1
Annealing conditions used in producing various grain sizes

| Material | Sintering temperature (°C) and time (h) | Grain size after sintering (μm) | Annealing temperature (°C) and time (h) | Grain size after annealing (μm) |
|---|---|---|---|---|
| 3Y-TZP | 1250 (0.5) | 0.2 | 1400 (1) | 0.47 |
| | | | 1500 (5) | 0.82 |
| | | | 1500 (30) | 1.51 |

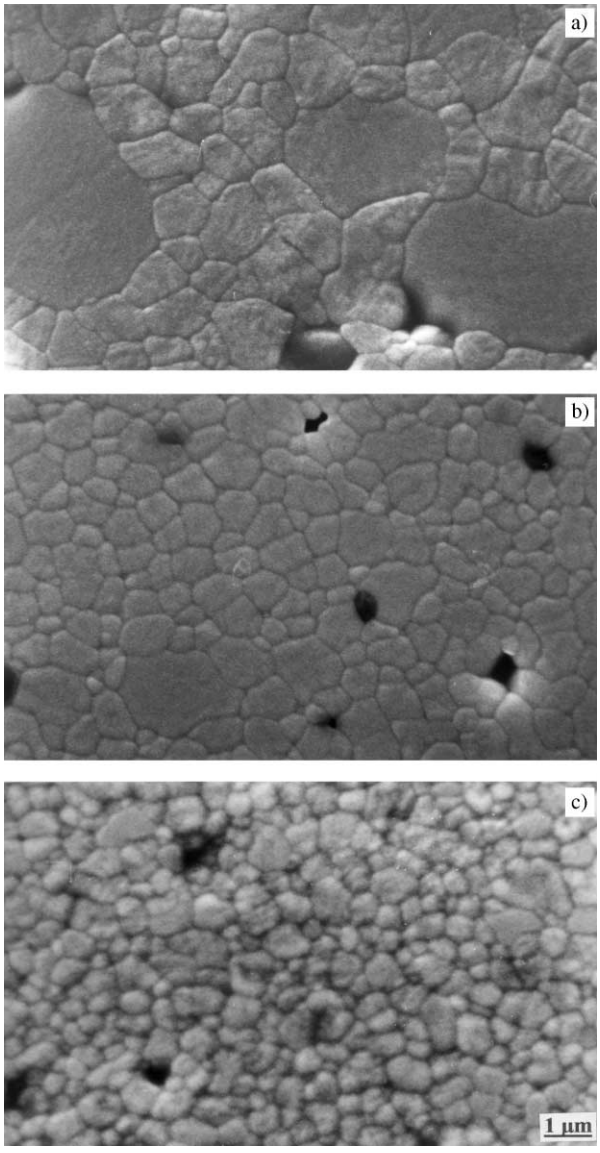


Fig. 1. SEM micrographs of a thermally etched surface of annealed samples with different grain sizes: (a) 1500 °C, 30 h, (1.51 μm), (b) 1500 °C, 5 h, (0.82 μm) and (c) 1400 °C, 1 h, (0.47 μm).

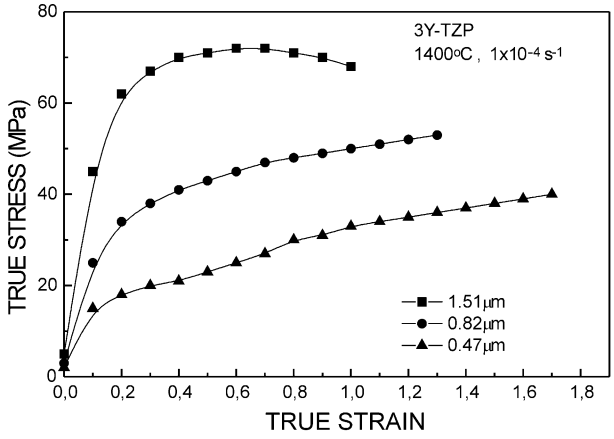


Fig. 2. True stress/strain curves for 3Y-TZP tested at 1400 °C and strain rate of $1 \times 10^{-4}$ $s^{-1}$ with various grain sizes.

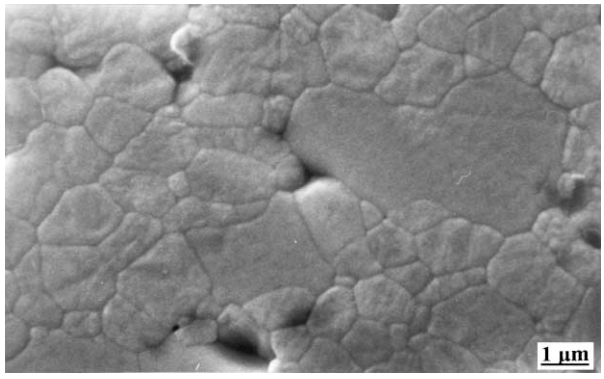


Fig. 3. SEM micrograph of 3Y-TZP annealed at 1500 °C for 30 h and tested to a predetermined strain of 125% at 1400 °C and $1 \times 10^{-4}$ $s^{-1}$. A bimodal distribution of small (original) and occasional large (grain growth) grains is evident. Tensile axis is normal to the specimen surface in the photograph.

True stress-true strain curves are plotted in Fig. 2 for 3Y-TZP with various grain sizes, tested at 1400 °C and at a strain rate of $1\times10^{-4}$ $s^{-1}$. It is evident that the value of elongation to failure increases with decreasing initial grain size and the flow stress increases with increasing grain size.

In polycrystalline materials, although grains are often equiaxed, they are rarely of equal size but generally show a log normal size distribution [11]. The nature of the grain size distribution has been shown to influence the stress/strain rate curve [12]. Materials which have a bimodal grain size distribution can be thought of as a mixture of two grain sizes that respond to stress differently, each being required to deform at the same strain rate. Large grains and small grains may deform by different mechanisms, e.g. at high stresses large grains may deform by dislocation creep and small grains by grain boundary sliding [13]. The overall $\ln\sigma$-$\ln\epsilon$ behaviour is governed by a combination of the behaviour of the two grain sizes. In the present study, the increased elongation to failure with decreasing grain size is probably a consequence of increased grain boundary sliding in the fine-grain materials and a result of decreased local stresses; in coarser grained materials, these stresses enhance cavitation cracking, thereby limiting the high temperature ductility. The results have shown that it is possible to attain high elongations even at grain sizes higher than those normally associated with superplastic behaviour in ceramics ($<1$ μm).

### 3.1. Effect of grain size on cavitation

The effect of initial grain size on cavitation was determined by straining the samples to a predetermined strain of 125% at a constant strain rate of $1\times10^{-4}$ $s^{-1}$ at 1400 °C. The low predetermined strain and test temperature were chosen to minimize strain-induced grain

growth and provide easily measurable levels of cavitation. It has been shown that dynamic and static grain growth are very severe at temperatures above 1400 °C in 3Y-TZP [14].

Cavities were observed in all specimens having grain sizes in the range of 0.47–1.51 μm after superplastic deformation and were frequently formed at occasional coarse grain boundary triple point junctions. This is shown in Fig. 3, which is a thermally etched cross-section of an annealed specimen with an average grain size of 1.51 μm. It was noted that there was very little grain growth under these conditions; an initial average grain size of 1.51 μm does not seem to have changed significantly. Fig. 4 shows internal cavities in three specimens with an initial average grain size of: (1) 1.51 μm, (2) 0.82 μm, and (3) 0.47 μm, respectively, elongated to a predetermined strain of 125% at $1\times10^{-4}$ $s^{-1}$ and 1400 °C. The photomicrographs were taken at approximately mid-points of the elongated specimen. It is seen that the level of cavitation increases with increasing initial grain size. It is also quantitatively apparent that a few large cavities exist in a dispersion of fine cavities and there is evidence in Fig. 4(a) of interlinkage of cavities in a direction essentially perpendicular to the tensile axis. This cavity interlinkage would cause a decrease in ductility and early fracture. By contrast, in Fig. 4(b) and (c), the cavities are uniform both in size and distribution, and there is no indication of cavity interlinkage.

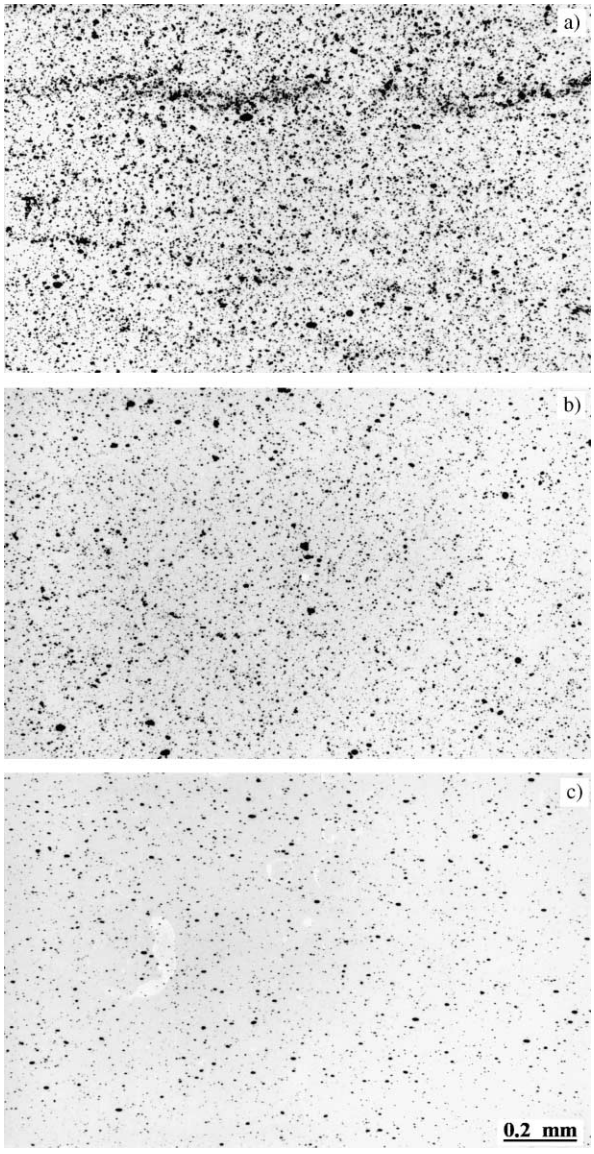


Fig. 4. Optical micrographs of 3Y-TZP with initial grain size of: (a) 1.51 μm, (b) 0.82 μm, and (c) 0.47 μm, elongated to a predetermined strain of 125% at $1 \times 10^{-4}$ $s^{-1}$ and 1400 °C. Tensile axis is horizontal.

The quantitative assessment of these observations, using an image analysis system, is presented in Fig. 5 (a) and (b), that show the number of cavities as a function of cavity size and circularity for 3Y-TZP specimens with various grain sizes, elongated to predetermined strain of 125% at a constant strain rate of $1\times10^{-4}$ $s^{-1}$ and at temperature of 1400 °C. It is seen in Fig. 5(a) that the number of cavities and cavity size increase as the initial grain size increases, reaching a value of 14% for the largest initial grain size material. This is consistent with many observations on superplastic metals [15] for which the general understanding is that accommodation involving diffusion and/or dislocation movement is

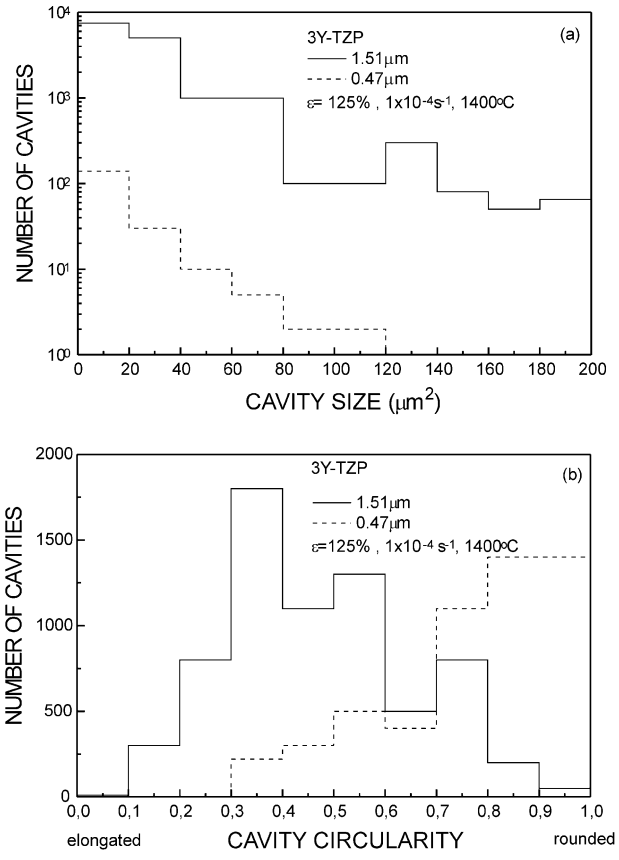


Fig. 5. Number of cavities versus (a) cavity area and (b) circularity for specimens tested to predetermined strain of 125% at $1 \times 10^{-4}$ $s^{-1}$ and 1400 °C.

required to maintain grain boundary cohesion. If accommodation is incomplete or slower than the grain displacement rate then cavitation will occur. The effect of grain growth or size is to make the accommodation process more difficult by increasing the unit path length for the operative mechanism. For 3Y-TZP tested here, the formation of cavities during high temperature deformation is dependent on initial grain size in addition to other factors including strain, strain rate, temperature and temperature regimen. To avoid a high level of cavitation which is frequently responsible for early fracture, a small stable grain size would be preferred.

The shape of individual cavities may be measured by defining a circularity coefficient as $4\pi$ Area/(perimeter)$^2$. There is a tendency, as shown in Fig. 5 (b), for cavities to become less rounded as grain size increases. This suggests that cavity interlinkage becomes more important at coarser grain sizes (the area density of cavities has a significant effect on tranverse interlinkage of cavities with the higher area density leading to early fracture), whereas at the finer grain sizes, the peak occurs at a value of around 0.8–1 which indicates that cavities are of a more rounded configuration.

## 4. Conclusion

The effect of grain size on superplastic deformation and cavity formation in 3 mol% yttria-stabilized zirconia polycrystal has been examined. It was seen that the value of elongation to failure increases with decreasing initial grain size and the flow stress increases with increasing grain size. Also the distribution of cavity size and cavity circularity have been quantitatively determined as a function of grain size. The results demonstrated that the amount of cavitation increase with increasing grain size.

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