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Nucleation and Growth Characteristics of Cavities during the Early Stages of Tensile Creep Deformation in a Superplastic Zirconia–20 wt% Alumina Composite

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Constant-stress tensile creep experiments on a superplastic 3-mol%-yttria-stabilized tetragonal zirconia composite with 20 wt% alumina revealed that cavities nucleate relatively early during tensile deformation. The number of cavities nucleated increases with increasing imposed stress. The cavities nucleate at triple points associated largely with an alumina grain, and then grow rapidly in a cracklike manner to attain dimensions on the order of the grain facet size. It is suggested that coarser-grained superplastic ceramics exhibit lower ductility due to the ease in formation of such grain boundary facet-cracks and their interlinkage to form a macroscopic crack of critical dimensions.

I. Introduction

The large elongations to failure associated with superplasticity are being utilized to form metallic components with complex shapes.[1] There has been a lot of interest in superplastic ceramics following the report of Wakai *et al.*[2] of an elongation of 170% in a 3-mol%-yttria-stabilized tetragonal zirconia (3 YTZ), and large strains have now been reported in numerous ceramics and ceramic composites.[3–8] Most of the studies have addressed the deformation behavior of such ceramics, and although the rate-controlling mechanism has not yet been identified unambiguously, especially in view of the sensitivity of these materials to small differences in trace impurity content,[8,9] it is accepted generally that grain boundary sliding is a major strain-contributing mechanism.

In contrast to the deformation behavior, there have been very few studies dealing with fracture of superplastic ceramics. The limited studies available deal mostly with an examination of cavitation at failure or at large elongations of >100%,[2,8,10–20] and there is very little information available on the development of cavitation during the early stages of superplastic deformation.

The present investigation was undertaken with the specific objective of characterizing the influence of stress and grain size on cavitation during the early stages of superplastic deformation.

II. Experimental Material and Procedures

The material chosen for this investigation was a fully dense 3-mol%-yttria-stabilized tetragonal zirconia composite reinforced with 20 wt% alumina. Tensile specimens of the composite, obtained from the Nippon Kagaku Co. in Japan, were tested under constant-stress tensile creep conditions, as described elsewhere.[21,22] Results of experiments conducted to determine the mechanical behavior indicated that the steady-state strain rate was described by a single constitutive relation over the entire range of conditions: $\dot{\epsilon} \propto \sigma^{2.8}\bar{L}_o^{-2.0}$, where σ is the applied stress and $\bar{L}_o$ is the linear intercept grain size.[22] The stress and grain size dependence of the creep rate is typical of superplastic flow in these materials.[3–9]

After the mechanical behavior of the composite was established, selected experiments were conducted to characterize the influence of stress and grain size on concurrent cavitation. Two specimens with an initial linear intercept grain size, $\bar{L}_o$, of 0.7 μm were tested at 1665 K and stresses of 9 and 96 MPa. The grains of both phases were equiaxed and of equal size. A local true strain of ~21% (measured from the reduction in cross-sectional area) was accumulated before the experiments were interrupted prior to specimen failure due to limitations in maintaining constant true stress to greater elongations. The influence of grain size on cavitation was examined by testing a specimen with initial zirconia and alumina grain sizes of 2.1 and 1.6 μm, respectively. At a temperature of 1665 K and a stress of 40 MPa the specimen failed at a true strain of 8.5%. The small strains accumulated prior to failure in the latter specimen are not superplastic; however, the creep characteristics are identical to specimens deformed superplastically. Therefore, it is expected that similar cavity nucleation and growth mechanisms operate under the various experimental conditions investigated and are representative of the early stages of cavitation in the superplastic ceramic composites.

After creep testing, the specimens were polished metallographically and examined by scanning electron microscopy. Quantitative data on the number density of cavities as well as the cavity area fraction were obtained using scanning electron micrographs exclusively in conjunction with an image analysis program. Sufficient micrographs were analyzed for each experimental condition such that greater than 150 cavities were measured. Electron transparent specimens were prepared using standard techniques and examined by transmission electron

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microscopy to obtain additional information on the evolution of cavitation damage and cavity morphology. Samples for electron microscopy were also prepared from undeformed material.

III. Experimental Results

(1) Qualitative Observations

Essentially similar features on the evolution of cavitation damage were observed under all experimental conditions. Cavities appeared to nucleate preferentially at triple points, often in conjunction with an alumina grain, as shown in the transmission electron micrograph (Fig. 1) obtained from a specimen with a grain size of 0.7 μm tested at 9 MPa to a true strain of 21%. In addition, as evident in Fig. 1, twinning was observed frequently within zirconia grains. However, twinning was also observed during the examination of the undeformed composite and therefore it is not anticipated that it is an important deformation mechanism under the conditions of this study. Cavity growth appeared to occur in a cracklike manner, until the cavity reached an adjacent triple point; this is illustrated in Fig. 2 by a transmission electron micrograph obtained from a specimen with a grain size of 2.1 μm tested at 40 MPa to a true strain of 8.5%. Figure 3 depicts the cracklike growth and interlinkage of cavities from adjacent triple points in a specimen tested at 96 MPa to a strain of 22%. Most of the cavities appeared to grow relatively easily to develop grain-facet-sized cracks. Such cracks were observed to be present predominantly in a direction perpendicular to the tensile stress axis, as shown in the scanning electron micrograph (Fig. 4) taken from the specimen tested at 40 MPa to a strain of 8.5%.

(2) Quantitative Data on Cavitation

All of the quantitative data obtained in the present study are represented in Fig. 5 as the variation with testing conditions in the number density of cavities (left axis) and cavitation area fraction (right axis). Inspection of Fig. 5 reveals that, for a grain size of 0.7 μm, an increase in stress from 9 to 96 MPa leads to an increase in the cavity density by a factor of ~6. This increase in the density of cavities is accompanied by a corresponding increase in the cavity area fraction, so that the ratio of the cavity area fraction to the number density, $\bar{a}$, remains essentially unchanged. A quantitative comparison indicates that the ratio $\bar{a}$ increases by a factor of ~3 with an increase in the grain size from 0.7 to 2.1 μm. It should be noted that the total

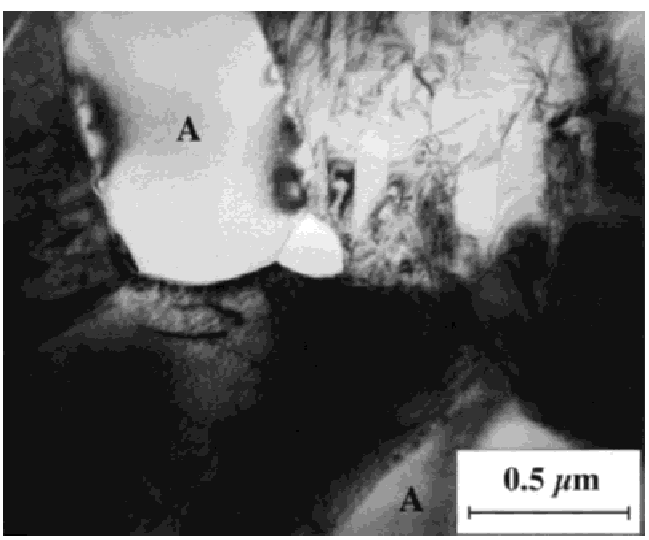

Fig. 1. Transmission electron micrograph illustrating cavity nucleation at triple points in a specimen with a grain size of 0.7 μm tested to a true strain of 21% at 9 MPa. Alumina grains are denoted by an ''A'' on the micrograph.

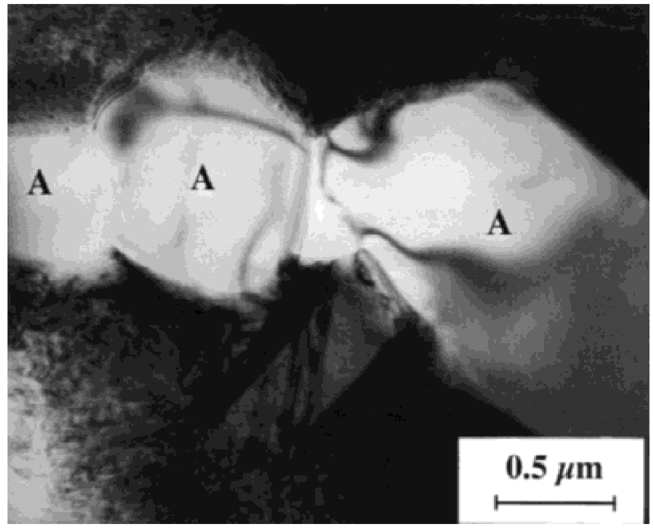

Fig. 2. Transmission electron micrograph illustrating cracklike cavity growth in a specimen with a grain size of 2.1 μm tested at 40 MPa to a true strain of 8.5%. Alumina grains are denoted by an ''A'' on the micrograph.

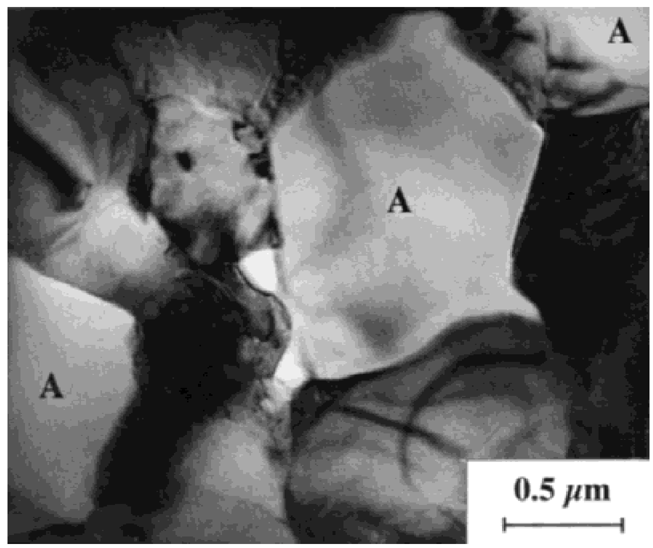

Fig. 3. Transmission electron micrograph illustrating cracklike cavity growth and interlinkage of cavities from adjacent triple points in a specimen with a grain size of 0.7 μm tested at 96 MPa to a true strain of 22%. Alumina grains are denoted by an ''A'' on the micrograph.

levels of cavitation recorded in this study are less than 1% area fraction. The presence of small and slightly varying amounts of monoclinic zirconia (of lower theoretical density) precludes the use of immersion techniques to accurately determine small cavitation levels in this material.

IV. Discussion

The present study provides the first set of experimental data on concurrent cavitation at low strains during the superplastic deformation of ceramics. It is interesting to note that an earlier study on the composite examined in this study indicated that cavitation levels of up to 30% were attained in specimens pulled to failure with elongations of >100%.[14]

(1) Cavity Nucleation

Experimental data on the evolution of cavitation damage with strain on specimens pulled to failure frequently appear to

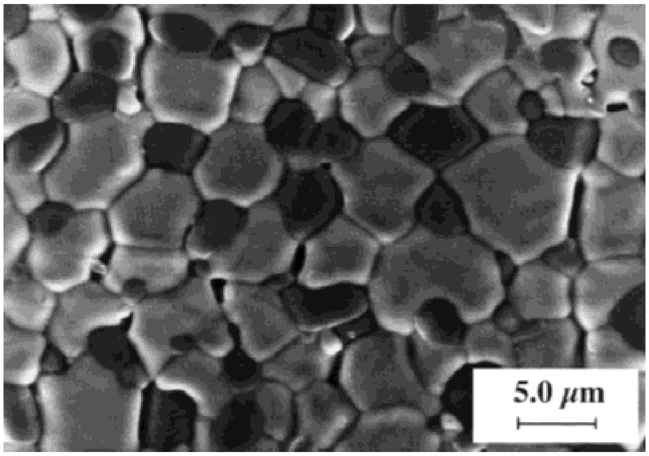

Fig. 4. Scanning electron micrograph illustrating the formation of grain boundary facet cracks in the specimen with a grain size of 2.1 μm tested at 40 MPa to a strain of 8.5%: the tensile axis is horizontal. The lighter phase is zirconia and the darker phase is alumina.

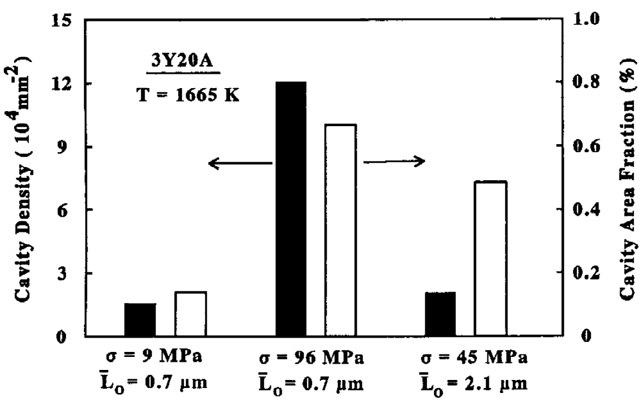

Fig. 5. Quantitative data illustrating the variation with testing condition in the number density of cavities (left axis) and the cavitation area fraction (right axis).

indicate that there is a significant threshold strain for cavitation.[14] However, the present microstructural and quantitative study indicates that concurrent cavitation commences at relatively low strains of <10%. The data obtained from specimens pulled to failure involve cavitation levels of up to 30%, which is substantially higher than the values of <1% recorded in the present study at low strains. Therefore, the apparent existence of a threshold strain is related to the scale of the axes used in the previous graphs and the accompanying simple curve-fit to the data: the data from the present study would appear to indicate near zero cavity area fraction if plotted on similar axes.

Cavities were observed to nucleate preferentially at triple points. This suggests that grain boundary or interphase ledges, if present, are not capable of sustaining the high stress concentrations necessary for cavity nucleation.[23] In addition, the cavities were associated frequently with an alumina grain. A comparison of creep data on alumina–zirconia composites by Chen and Xue[6] suggests that pure alumina and zirconia exhibit similar creep rates. However, as noted by Wakai *et al.*,[24] the addition of a small quantity of zirconia to alumina leads to a significant retardation of the creep rate. Therefore, following Chen and Xue,[6] it is suggested that alumina with a small quantity of zirconia is much more creep resistant than alumina. Consequently, the alumina particles act as the hard phase in the zirconia–alumina composite, so that cavities are nucleated in association with hard alumina particles and grain boundary sliding. It is interesting to note that in a recent study on an alumina–silicon carbide composite, cavities were associated with SiC particles which acted as a hard phase.[25]

The influence of grain size on cavity nucleation could not be assessed directly because the data at a grain size of 2.1 μm were obtained at a different stress than those for a grain size of 0.7 μm. The difference in stress utilized in this study reflects an experimental difficulty in testing coarser-grained specimens; at higher stresses, the specimens fractured almost instantaneously, whereas at lower stresses the creep rates obtained would be too low for a laboratory study.

(2) Cavity Growth

The general process of cavity growth by a diffusion process involves vacancy diffusion along a grain boundary to the tip of a cavity, and the subsequent incorporation of the vacancy into the cavity by surface diffusion; grain boundary and surface diffusion are sequential processes and the slower one will control cavity growth. When cavity growth is controlled by grain boundary diffusion, the cavities adopt a quasi-equilibrium shape; on the other hand, when cavity growth is controlled by surface diffusion, the cavities will adopt a cracklike morphology.[26] The experimental observations recorded in the present study indicate that cavities adopt a cracklike profile, and they grow along a grain boundary in a direction perpendicular to the tensile axis. Such observations are consistent with a surface-diffusion-controlled cavity growth process, as modeled earlier by Chuang and Rice.[26] It is interesting to note that Porter *et al.*[27] made similar observations during the creep of polycrystalline alumina. Unfortunately, the lack of diffusion data for the system precludes a quantitative comparison of the experimental observations with theoretical predictions.

The experimental data on cavitation at low strains suggest that cavities grow by a diffusion-related process. However, a recent study by Ma and Langdon[19] on cavitation in zirconia specimens pulled to large elongations concluded that cavity growth occurred by a plasticity-controlled process. This dichotomy can be resolved possibly by comparison with observations on superplastic metallic alloys. Diffusion- and plasticity-controlled growth processes operate independently so that the faster one is rate controlling. Early studies on superplastic metallic alloys indicated that small cavities grow by a quasi-equilibrium diffusion process, whereas large cavities grow by a process involving the plastic deformation of material surrounding a cavity.[28,29] In accordance with such analysis in metallic alloys, it is suggested that small cavities in the composite grow by a surface-diffusion-controlled process, whereas larger cavities may grow by a plasticity-controlled mechanism.

The quantitative observation that the ratio $\bar{a}$ scales with the grain size implies that cavity growth is limited by the grain boundary facet length, such that cavities nucleated at triple points grow readily to become grain boundary facet cracks.

(3) Implications for Ductility in Superplastic Ceramics

Chen and Xue[6] compiled data on ductility in superplastic ceramics, and they noted that the flow stress was the primary factor governing the ductility of ceramics. Kim *et al.*[17] also analyzed the tensile ductility of superplastic ceramics in terms of the Zener–Hollomon parameter $Z\,[=\dot{\epsilon}\exp(Q/RT)]$, where $\dot{\epsilon}$ is the strain rate, Q is the activation energy, R is the gas constant, and T is the absolute temperature. Both of these approaches are essentially identical because the flow stress at elevated temperatures is related to the strain rate and temperature by an expression of the form $\dot{\epsilon} \propto \sigma^n \exp(-Q/RT)$, where n is the stress exponent. In addition, Kim *et al.*[17] utilized a fracture mechanics type approach to rationalize the grain size dependence of the ductility in superplastic ceramics.

The present experimental observations provide a more microstructurally based explanation for the significant decrease in ductility with an increase in grain size. Fracture in superplastic ceramics is controlled by the formation of a macroscopic transverse crack with a critical dimension, which entails the interlinkage of the transverse grain boundary facet-sized cavities along interfaces that are inclined to the tensile axis. For a specimen with a given cross-sectional area, there are fewer such inclined grain boundary facets for coarser-grained specimens and the individual facets are larger, so that the formation of a critical transverse crack occurs more easily in such specimens compared to fine-grained specimens. A probabilistic approach adopting realistic grain size distributions and shapes is necessary to quantify such a process.

V. Summary and Conclusions

Constant-stress tensile creep experiments were performed on a superplastic 3-mol%-yttria-stabilized tetragonal zirconia composite with 20 wt% alumina to study the early stages of cavitation during superplastic deformation. Microstructural inspection revealed that cavities nucleated at triple points associated frequently with alumina particles, and grew rapidly, perpendicular to the tensile stress axis, in a cracklike manner to develop grain boundary facet dimensions. Quantitative measurements revealed that the number density of cavities increased with increasing stress, although the average cavity size remained constant. The average cavity size also scaled with the grain size. These results indicated that cavity growth is limited by the grain boundary facet size, and the early failure of coarse grain ceramics is related to the rapid development of transverse grain boundary facet cracks and their interlinkage to form a macroscopic crack.

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