Cavitation failure during superplastic tensile loading in alumina-base ceramics

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Abstract. Tensile failure in superplastic alumina-base materials shows a close similarity to ductile failure in metallic materials containing voids or hard inclusions. The occurrence of microcracks, which is followed by macroscopic cracking towards final failure, cannot be explained from the brittle propagation of preexistent defects, but from the coalescence of intergranular cavities formed and grown by superplastic deformation. The cavity coalescence is shown to occur as the cavity separation distance decreases to a certain level owing to an increase in the number and size of the cavities.

1. Introduction
Although earlier studies on superplastic ceramics have often reported that tensile failure occurs through cracking in a direction perpendicular to the stress axis [1-7], the mechanisms of such cracking have still been unclear. For example, the causes of cracking assumed in the studies are essentially different from each other. Kim et al. [1] proposed that cracking is caused by the brittle propagation of preexistent defects and the propagation occurs from the beginning of tensile deformation. From microstructural features after deformation, Schissler et al. [2] and Chokshi et al. [3] associated the cracking with extensive interlinkage among adjacent cavities. There have, however, been few quantitative studies on these assumptions and on the details of tensile failure in superplastic ceramics. The present study was undertaken to examine this issue by using alumina-base materials under different combinations of the initial grain size, tensile rate and deformation temperature. Emphasis was placed on the onset conditions of cracking.

2. Experimental methods
The following materials were synthesized from high purity oxide powders (> 99.97%) by methods described elsewhere [5]: a monolithic alumina doped with 0.2 wt% MgO and a composite alumina dispersed with 10 vol% ZrO2 particles. For relative densities larger than 99%, the grain size of the alumina phase (defined as 1.56 times the average intercept length) was adjusted to \(d_0 = 0.7-2.8 \, \mu m\) and \(0.45-2.0 \, \mu m\) for the MgO-doped and the ZrO2-dispersed materials, respectively. From these materials, flat tensile specimens with gauge dimensions of \(l_0 = 10-3, w_0 = 3.5 \, mm\) were machined and pulled to failure or to prescribed strains with an Instron-type machine. The ranges of the loading temperature and the strain rate were 1400-1550 °C and 2×10^{-4} - 3×10^{-3} s^{-1}, respectively. After loading, a surface layer of 300-500 \, \mu m in thickness was removed from the gauge portion for polishing. By using quantitative optical microscopy [5] of which resolution limit was set to 0.5 \, \mu m, the area-equivalent circle diameter, the major and minor axis lengths and the aspect ratio (the major to the minor axis ratio) of each cavity and the volume fraction of cavity damage were measured at the center of the as-polished gauge portion.
study on cavity growth in the present materials [7] showed that cavity damage in the present materials was similar for a given value of increasing strain, showing continuous cavity formation and growth with loading. The evolution of measurement was made. The size distribution shifts towards higher densities and larger sizes with increasing strain. The interval of cavity size is 0.8 μm.

3. Examination of the brittle crack-propagation model

Figure 1 summarizes the tensile ductility of the present material as a function of the average flow stress, $σ_f/ε_f$, where $σ_f$ is the stress at a strain of $ε_f$ and $ε_f$ is the failure strain. There is a trend of decreasing tensile ductility with increasing flow stress. Such a trend that has often been noted for superplastic ceramics [1-4] is reasonable since higher stresses may generally accelerate primary processes of failure. However, tensile ductility shows a large scatter for a given flow-stress level that can be attained by different combinations of the grain size and the loading conditions. Thus, the data show that tensile ductility cannot be related simply to the flow-stress level.

Figure 1 also indicates that the tensile failure of the present materials cannot be explained from the brittle crack-propagation model [1] given by equation (1).

$$ε_f = \ln \left( \frac{(2γ_s - γ_gb)}{πC_0} \right)^{1/q} \sigma^{-2q},$$

where $ε_f$ is the fracture strain, $γ_s$ is the surface energy, $γ_gb$ is the grain-boundary energy, $E$ is the elastic modulus, $C_0$ is the initial crack size related to the initial grain size and $q$ is the rate constant of a crack-growth law given by $dC/dε = qC$ and it takes a value of about 4 [1]. For a given chemical composition, namely a given combination of $γ_s$, $γ_gb$ and $E$, Eq. (1) predicts a linear relationship between $ε_f$ and $ln σ$ with a slope of $-2/q$. However, the $ε_f$-$σ_f$ data given in figure 1 show a large scatter on the logarithmic scale. A $q$-value of about 4 that appears for a certain set of the data does not support the brittle cracking model. A study on cavity growth in the present materials [7] showed that $q$ is smaller than 2.0 except for a last stage of deformation where the tensile strain exceeds 90% of the failure strain. This fact is contrary to such a premise of the model [1] that steady crack growth with $q \sim 4$ occurs from the beginning of loading.

4. Evolution of cavitation damage relating to tensile failure

Figure 2 shows the evolution of cavity size distribution as a function of the local strain, $ε_l$, at which the measurement was made. The size distribution shifts towards higher densities and larger sizes with increasing strain, showing continuous cavity formation and growth with loading. The evolution of cavity damage in the present materials was similar for a given value of $ε_l/ε_0$, where $ε_0$ is the local strain at the failed tip. For $ε_l/ε_0 \sim 0.9$, cavity coalescence was limited or localized and most cavities were rather equiaxed with diameters of $< 10 \, \mu m$ and aspect ratios of $< 2.0$. Microcracks (figure 3(a)), which
were defined as cavities having major axis lengths of $\geq 20$ $\mu$m and aspect ratios of $\geq 2.0$ in this study, did not appear until the value of $\varepsilon / \varepsilon_f$ exceeded $\sim 0.9$. For $\varepsilon / \varepsilon_f > \sim 0.95$, the size and the number of microcracks increased steeply, and some of them interlinked or grow in the direction normal to the tensile axis, resulting in macroscopic cracks for final failure. Such a close relationship between microcracking and tensile failure is explicitly confirmed in figure 3 (b), where the failure strain is proportional to the onset strain of microcracking with a slope of about 0.9.

5. Microcracking through cavity coalescence

We examined the onset of microcracking using a model for cavity coalescence through necking of the matrix among adjacent cavities [8], since the microstructural observation strongly suggests that this mechanism may work in the present material (figure 3(a)). The model predicts that cavity coalescence, namely microcracking occurs when the following condition is fulfilled:

$$P (1 - f) = \frac{\sigma_m}{Y} + \frac{3 + \nu}{3\sqrt{\nu^2 + 3}}$$

(2)

where $P$ is the plastic constraint factor, $\sigma_m$ is the mean normal stress, $Y$ is the uniaxial yield stress of the matrix and $\nu$ is the Lode variable and $f$ and $f_0$ are the current and the initial volume fractions of cavities, respectively. In the present materials, $f_0$ is in a range of $6 \times 10^{-4}$ - $2 \times 10^{-3}$. The plastic constraint factor can be calculated from cavity data as $P = 0.1/(D_{av}/\lambda)^2 + 1.2/(D_{av}/L_{av})$/$^2$, where $D_{av}$ and $L_{av}$ are the average diameter and the center to center spacing of cavities, respectively, and $\lambda = L_{av} - D_{av}$ is the cavity-separation distance. The equation shows that the $P$-value decreases with increasing cavity number and size.

As typically shown in figure 4, there is a close relationship between the evolution of the plastic constraint factor and the onset of microcracking. That is, microcracking starts to occur as the value of $P$ decreases from the initial range of 209-216 to a narrow range of 2.1-2.9 owing to an increase in the number and size of cavities (figure 2). This relationship was found both in the MgO-dope and in the ZrO$_2$-dispersed materials independently of the combination of the initial grain size, temperature and strain rate. From this result, we examined equation (2) as follows. Assuming the isotropic growth of spherical cavities as a first-order approximation, and considering that little necking occurs in the gauge portion of superplastic ceramics and $\nu$ is unity for uniaxial tension, equation (2) is simplified as [9]:

$$P (1 - f) \approx 1.$$  

(3)

The values of $P(1 - f)$ at the onset of microcracking were calculated by using the cavity-size distributions and $f = 0.11-0.33$. The calculated values are plotted in figure 5 as a function of the failure strain, $\varepsilon_f$. 

**Figure 3** (a) Microcracking observed in the ZrO$_2$-dispersed material loaded at 1500 °C and at $2 \times 10^{-4}$s$^{-1}$ to 0.93$\varepsilon_f$ and (b) the relationship between the onset strain of microcracking and the failure strain.
2. Microcracking, which appears as the strain exceeds \( \varepsilon_\text{f} \sim 0.9 \), decreases to a certain level owing to an increase in the number and size of cavities. Analysis of cavitation data shows that the microcracking starts to occur as the cavity-separation distance

Figure 4 Typical evolution of the plastic constraint factor as a function of strain. The arrows indicate the onset points of microcracking.

Figure 5 Plastic constraint factor for the onset of microcracking as a function of the failure strain.

Figure 5 indicates that the cavity-coalescence model works well for the onset of microcracking in the present materials. The value of \( P(1-\varepsilon) \) is decreased from the initial range of 116-227 to a narrow range of 1.9 ± 0.3 (MgO-doped) and 1.7 ± 0.2 (ZrO\(_2\)-dispersed) at the onset of microcracking, independently of the combination of the initial grain size, strain rate, temperature and failure strain. The \( P(1-\varepsilon) \) values slightly larger than unity at the onset of microcracking may arise mainly from the simplification of the cavity shapes. The result shown in figure 5 is in contrast to the large scatter appearing in the \( \varepsilon_\text{f} - \sigma_\varepsilon \) relationship (figure 1) and leads to a conclusion that the primary process of tensile failure in the superplastic alumina is cavity coalescence due to internal necking of the matrix among adjacent cavities.

6. Conclusions
1. Tensile ductility in superplastic alumina cannot be explained only from the level of flow stress: failure strain \( \varepsilon_\text{f} \) shows a large scatter for a given flow stress level attained by different combinations of the strain rate, loading temperature and grain size.
2. Microcracking, which appears as the strain exceeds \( \sim 0.9\varepsilon_\text{f} \) and leads to macroscopic cracking for final failure, does not occur through a brittle process, but through a plastic process of cavity-coalescence. Analysis of cavitation data shows that the microcracking starts to occur as the cavity-separation distance decreases to a certain level owing to an increase in the number and size of cavities.

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