Compressive Deformation of Y_2O_3 -Stabilized ZrO_2/Al_2O_3 Composite

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Y_2O_3 -安定化 ZrO_2/Al_2O_3 複合体の圧縮変形

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Compressive deformation of 80 wt% ZrO_2 (3 mol% Y_2O_3)/20 wt% Al_2O_3 composite was studied in air at temperatures from 1400° to 1550°C. Extraordinary large deformation (up to -1.8) was observed at strain rates from 10^{-3} to 10^{-4} s $^{-1}$. The stress exponent was from 1.2 to 2 and activation energy was 620 $\pm$ 40 kJ/mol. It was observed that the Al_2O_3 aggregate evolved during the deformation. This phenomenon was explained by the grain rearrangement model as a consequence of grain switching during the superplastic flow.

[Received July 1, 1986]

Key-words : ZrO_2/Al_2O_3 composite, TZP, Alumina, Compression test, Superplasticity, Grain boundary sliding

1. Introduction

It is well known that many two-phase alloys show superplasticity¹⁾. The research on superplastic multi-phase ceramic composites would provide basis both for understanding the mechanism²⁾ and for finding possibilities of engineering applications in ceramic industry.

The authors³⁾⁻⁵⁾ have found the superplasticity in Y_2O_3 -stabilized tetragonal ZrO_2 polycrystals (Y-TZP) at the temperature range of a two-phase field of tetragonal-cubic mixtures in the ZrO_2 - Y_2O_3 system. Katsumura and Fukuhara⁶⁾ reported the enhanced ductilities in Al_2O_3 -Ti(C_x , N_{1-x}) ceramics the mechanism of which they named "heterogeneous grain boundary sliding".

It is of scientific interest to compare the deformation mechanism of ZrO_2/Al_2O_3 composite which has a two-phase duplex microstructure with that of quasi-single phase ceramics, as TZP and Al_2O_3 .

In this report, deformation properties and possibility for superplasticity of ZrO_2/Al_2O_3 have been studied by compression tests.

2. Experimental procedure

The ZrO_2/Al_2O_3 composite was fabricated from

80 wt% ZrO_2 which contained 3 mol% Y_2O_3 in solid solution and 20 wt% Al_2O_3 powders. The pressed compact was sintered in air and then hot isostatic pressing was performed in Ar gas^{7),8)}.

The density of the composite was 5.49 g/cm³. The average grain-sizes for the single-phase particles were approximately 0.9 μ m for ZrO_2 and 0.8 μ m for Al_2O_3 . The bending strength was from 1800 to 2000 MPa at a room temperature.

Uniaxial compression test specimens (10 mm in height) were prepared by putting two rectangular parallelepipeds with dimension of 7 $\times$ 7 $\times$ 5 mm one upon another. Compression tests were carried out in air at temperatures up to 1550°C by using an Instron universal testing machine. The stress was transmitted to the specimen by two SiC rods at cross-head speeds of 0.05 to 1 mm/min. Load-strain curves were obtained by assuming that the reduction in length of the specimen corresponded to the displacement of the cross-head.

3. Results

3.1 Flow behavior

True stress (σ) vs. true strain (ϵ) curves at different initial strain rates at 1500°C are shown in

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Fig. 1. The true strain rate, $\dot{\epsilon}$, increased with the reduction in length of the specimen under a constant cross-head speed. The increase in flow stress at large strains is due to the change of an effective strain rate.

The value of true stress at a true strain of -0.2 in Fig. 1 was read and plotted as a function of the strain rate in Fig. 2. The flow behavior is given by the following equation,

$$\sigma = K \cdot \dot{\epsilon}^m, \quad (1)$$

where K is a constant and m is a strain rate sensitivity index. The m -value is equal to $1/n$, where n is a stress exponent. The stress exponents were calculated from the slope of the lines in Fig. 2 by using the least-square method. The plot yielded

$$n = 1.5 \pm 0.4 \quad (2)$$

at the temperature range from 1400° to 1550°C .

The strain rate is described by an equation of the following form,

$$\dot{\epsilon} = A \sigma^n \exp(-\Delta Q/RT) \quad (3)$$

where ΔQ is the activation energy, R is the gas constant, and T is the temperature. The strain rates, $\dot{\epsilon}$, at three values of stress, 10, 20 and 40 MPa were obtained from Fig. 2 and plotted against the $1/T$, as is shown in Fig. 3. The

Fig. 1. True stress against true strain curves at different initial strain rates ($\dot{\epsilon}_0$) at 1500°C .

Fig. 2. Strain rate dependence of the flow stress at various temperatures.

Fig. 3. Temperature dependence of the strain rate at three values of stress.

activation energies were calculated from the straight lines to be,

$$\Delta Q = 620 \pm 40 \text{ kJ/mol} \quad (4)$$

3.2 Microstructure

Figure 4 shows the evolution of microstructure after the compressive deformation. The grain growth during the deformation was clearly observed. The as-sintered specimen shows equiaxed Al_2O_3 grains ($\approx 0.8 \mu\text{m}$) dispersed uniformly among the ZrO_2 grains ($\approx 0.9 \mu\text{m}$). The elongated grains were observed in the cross-section of the deformed specimen which was strained until $\epsilon_t = -1.8$. The longitudinal dimension ($d_{\parallel}$) and transverse ($d_{\perp}$) dimension of the grains were measured. The dimension $d_{\parallel}$ of the grains was approximately $1.6 \mu\text{m}$ for ZrO_2 and $1.3 \mu\text{m}$ for Al_2O_3 . The grain aspect ratios (GAR) of $d_{\parallel}$ to $d_{\perp}$ of ZrO_2 and Al_2O_3 grains were 2 and 1.7, respectively.

If a cube-shaped grain is distorted during deformation so that the length and breadth are $d_{\perp}$ and $d_{\parallel}$, respectively. The intragranular strain, ϵ_g , is given by

$$\begin{aligned} \epsilon_g &= \ln(d_{\parallel}/(d_{\perp}d_{\parallel}^2)^{1/3}) \\ &= 2/3 \ln(d_{\parallel}/d_{\perp}) \end{aligned} \quad (5)$$

The total strain, ϵ_t , may be expressed as¹⁰⁾

$$\epsilon_t = \epsilon_g + \epsilon_{\text{gbs}}, \quad (6)$$

where ϵ_{gbs} is the strain contributed by grain boundary sliding. The measured value of intragranular strain, -0.4 , gives the contribution of the grain boundary sliding to the total strain to be, $\epsilon_{\text{gbs}}/\epsilon_t = 75\%$.

Cavities at triple grain junctions were also observed in the deformed specimens. Cavities were found to be generated at Al_2O_3 - Al_2O_3 boundary and Al_2O_3 - ZrO_2 boundary, but not observed at ZrO_2 - ZrO_2 interfaces. Also it was observed that

Fig. 4. (a) SEM micrograph of the as-sintered composite. Al_2O_3 is dark-phase. (b) The deformed specimen ($\epsilon_1 = -1.8$) strained at 0.05 mm/min at 1500°C . Arrows indicate the compression direction.

the agglomerate of Al_2O_3 grains evolved during the deformation and aligned perpendicularly to the compression direction.

4. Discussion

The fact was clearly demonstrated that extensive grain boundary sliding (GBS) occurred during the compressive deformation of $\text{ZrO}_2/\text{Al}_2\text{O}_3$ composite just as during the superplastic flow of Y-TZP. There are three types of grain boundary in the two-phase microduplex $\text{ZrO}_2/\text{Al}_2\text{O}_3$ composite, namely, ZrO_2 - ZrO_2 boundary (Z-Z), ZrO_2 - Al_2O_3 boundary (Z-A), and Al_2O_3 - Al_2O_3 boundary (A-A). If the content of Al_2O_3 grains is small, the deformation property of the composite will be similar to that of TZP. Inversely, if the content of Al_2O_3 grains is large, the deformation property of the composite will be similar to that of Al_2O_3 ceramics.

The macroscopic strain rate of 3 mol% Y_2O_3 -stabilized TZP was expressed by the following equation⁴⁾,

$$\dot{\epsilon}(\text{s}^{-1}) = 2 \times 10^5 \exp(-380000(\text{J/mol}) / RT) \sigma^{2.1} / d^{1.8}, \quad (7)$$

where the unit of stress, σ , is [MPa], and that of grain size, d , is [μm].

Venkatachari and Raj¹¹⁾ found that the compression creep property of MgO-doped fine-grained alumina (grain size $\approx 1.6 \mu\text{m}$) at 1420°C was consistent with the equation for Coble creep.

Figure 5 shows the flow stress vs. strain rate plots for the composite and single-phase materials with different grain sizes. The flow stress of the

Fig. 5. Flow stresses of the two-phase microduplex structure ceramic ($\text{ZrO}_2/\text{Al}_2\text{O}_3$ composite) and quasi-single phase ceramics (TZP, Al_2O_3) at 1420°C .

composite was higher than those of Al_2O_3 and TZP which had an average grain size of $1 \mu\text{m}$. The value of stress exponent of the composite, $n = 1.5 \pm 0.4$, was intermediate between that of Al_2O_3 ($n=1$) and that of TZP ($n=2$).

In order that Al_2O_3 aggregates are evolved, it is necessary for dispersed fine Al_2O_3 grains to be contacted with one another. This evolution can be explained by assuming that grain switching occurs during the deformation by grain boundary sliding, as is shown in Fig. 6. Initially separated Al_2O_3 grains, A1 and A2 in Fig. 6, will be contacted with each other just like as Ashby-Verrall model¹²⁾ for superplasticity. If grain boundary sliding between Al_2O_3 grains does not take place easily when compared with other types of grain boundary sliding, (Z-Z $\approx$ Z-A $>$ A-A), contacted Al_2O_3 aggregate will not be separated. Then the growth of the aggregate occurs as the

Fig. 6. A model for evolution of Al_2O_3 aggregate by grain switching.

deformation proceeds. Furthermore, grain switching that is not accompanied with the separation of the Al_2O_3 grains seems to cause grain rearrangement of the aggregate perpendicular to the compressive stress (Fig. 6(d)).

5. Conclusion

Extraordinary large strain (-1.8) was obtained at strain rates of 10^{-3} to 10^{-4} s^{-1} in compression test of $\text{ZrO}_2/\text{Al}_2\text{O}_3$ composite which had microduplex structure. Microstructural evolution during the deformation suggested the grain switching mechanism for grain rearrangement that was very similar to Ashby-Verral model for superplasticity. Cavitations observed at Al_2O_3 - Al_2O_3 boundaries and Al_2O_3 - ZrO_2 boundaries were not so significant. Superplastic elongation can be expected for fine-grained

$\text{ZrO}_2/\text{Al}_2\text{O}_3$ composite.

Acknowledgement The composite was kindly provided by Y. Kubota of Toyo Soda Manufacturing Co., Ltd. The authors would like to thank Dr. K. Hayakawa for his helpful discussions.

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