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Colloidal Processing and Superplasticity of Monoclinic-, Tetragonal-, and Cubic-zirconia Dispersed Aluminas

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High temperature tensile ductility in alumina-based ceramics is usually limited due to dynamic grain growth. In this study, superplasticity in zirconia dispersed alumina has been achieved using colloidal processing, using monoclinic, tetragonal and cubic zirconia powders. Colloidal processing helps to prevent the agglomeration of particles and enables particle dispersion to be controlled in powder processing. The dispersion of particles in the suspensions was stabilized by electrosteric repulsion, and caused by the adsorption of added polyelectrolyte ionized onto the particle surface. The suspensions were consolidated by slip casting, followed by cold isostatic pressing and sintering in air at 1673K for 2h. Differences in static alumina grain growth in the different zirconia-dispersed alumina composites were caused by differences in the distribution of the zirconia particles. The tensile ductility of these materials was found to depend on the kind of zirconia used. Limited superplasticity seems to be caused by dynamic alumina grain growth during deformation and residual defects. Those composites with reduced grain size and fewer residual pores had improved tensile ductility.
Key words: superplasticity, alumina, zirconia, colloidal processing, grain growth

1. INTRODUCTION

It is well known that a dispersion of zirconia in alumina is a very effective method of improving the fracture toughness [1]. One of the major problems limiting the practical use of this material is its poor workability.

The high-temperature tensile ductility of undoped, fine-grained alumina is limited to an engineering strain of less than 20% because of rapid dynamic grain growth. Many studies have attempted to improve the tensile ductility in alumina-based ceramics. However, the tensile ductility of alumina, for which dynamic grain growth is suppressed by MgO, is limited to 80% [2-5]. An alumina-30 vol% spinel composite with a large tensile elongation of 396% has been obtained by Takigawa et al [6].

Zirconia particle dispersions were very effective in inhibiting grain growth, but the increase in flow stress due to the suppression of grain boundary sliding limited the tensile ductility [5, 7-9]. A reduction in the initial grain size of zirconia dispersed alumina is necessary for improving flow stress and enhancing tensile ductility.

Using fine particles as starting materials is a convenient way to improve the final microstructure. However, fine particles tend to spontaneously agglomerate due to van der Waals forces. The agglomerated particles form large pores, and high sintering temperature is necessary to eliminate the pores and form dense bodies. Colloidal processing helps to prevent the agglomeration of fine particles and enables particle dispersion to be controlled during powder processing. The authors have reported that a 10 vol% zirconia dispersed alumina with a homogeneous and fine microstructure can be produced through colloidal processing and which is capable of large tensile elongation exceeding 500% [10].

The lattice structure of zirconia depends on the amounts and kinds of stabilizing elements used, such as Y_2O_3 and CeO_2 [11, 12]. In this study, starting materials for dispersion were monoclinic-, tetragonal-, or cubic-zirconia. Thus the effects of different kinds of zirconia on the microstructure and tensile properties have been investigated.

2. EXPERIMENTAL

2.1 Powder

The starting materials were high-purity (>99.99%) α-alumina powder (Taimei Chemicals, TM-DAR) with particles averaging 0.2 μm and four different zirconia powders (Tosoh. Co. Ltd.). TZ-0, TZ-3Y, TZ-8Y, and TZ-12CE are undoped monoclinic zirconia, 3mol% yttria-stabilized tetragonal zirconia, 8mol% yttria-stabilized cubic zirconia, and 12mol% cerium oxide-stabilized tetragonal zirconia, respectively. The particle sizes were 0.075, 0.062, 0.076, and 0.094 μm, respectively.

2.2 Colloidal processing

Aqueous suspensions containing 30 vol% solids of alumina containing 15 vol% and 10 vol% zirconia were prepared. When suspensions consist of particles of different size and density, segregation occurs because of differences in the sedimentation rate during slip casting [13, 14]. Segregation has been shown to be inhibited by hindering sedimentation if the solid content exceeds 30 vol% in suspension [15].

Electrosteric stabilization was used to disperse the fine particles and was achieved by polyelectrolyte adsorption on the surface of the particles. The adsorbed polyelectrolyte is ionized, and the negative charge on the surface is sufficiently large to cause stable particle dispersion due to strong electrostatic and steric repulsion. Polyelectrolyte (poly(ammonium acrylate), Toaghosei Co., ALON A-6114) was added to the suspensions [16, 17]. In addition, re-dispersion is needed to properly disperse both fine particles in suspensions because of spontaneous agglomeration. The authors have reported the effect of ultrasonication on microstructures produced by colloidal processing [18]. Therefore, suspensions were prepared by stirring with sufficient ultrasonic treatment to obtain a uniform dispersion of zirconia particle. The suspensions were evacuated in a desiccator to eliminate air bubbles, then consolidated by slip casting. The green compacts were densified by cold isostatic pressing (CIP) at 400 MPa, and the sintering at 1673K for 2 h.

2.3 Characterizing and testing

The rheological characteristics of the suspensions were measured by a cone and plate viscometer. Densities of the green and sintered bodies were measured by the Archimedes' method using kerosene. The microstructure of the sintered specimens was observed by SEM on polished and thermally etched samples. The mechanical properties of sintered samples with a gage length of 10mm were examined by tensile testing at 1773K using an initial strain rate of 1.7×10^{-4} s^{-1}.

3. RESULTS AND DISCUSSION

The rheological characteristics are a measure of the powder dispersion in the suspensions. Plots of the apparent viscosity versus shear rate for the suspensions consisting of alumina and each kind of zirconia are shown in Fig. 1. Cleanly, the apparent viscosity depends on the kind of zirconia powder. The apparent viscosity of suspensions increases, for an increase in the interaction between particles due to agglomeration. The suspension consisting of TZ-8Y was dispersed better than the other suspensions, since the apparent viscosity is the lowest.

Figure 2 shows the relative densities of the green compacts formed by slip casting and CIP treatment. The relative density of the TZ-8Y dispersed compact prepared from the well-dispersed suspension is the highest. The density of the TZ-3Y dispersed compact prepared from the suspension with the largest viscosity was relatively low. The densities of all the compacts produced by slip casting and followed by CIP treatment was more than 60%. These densities are higher than that produced by the compaction of conventional uniaxial pressing.

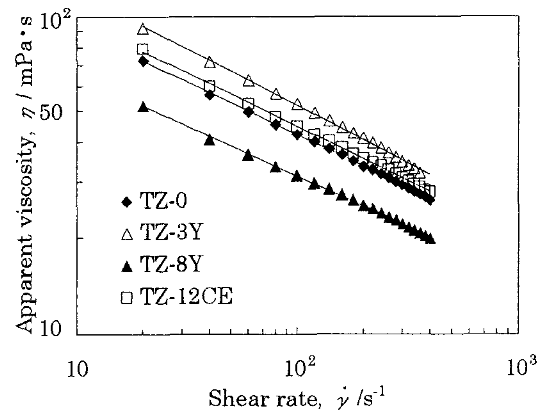

Fig. 1 Effects of the kind of zirconia particles on the apparent viscosity of the suspension containing alumina – 15 vol% zirconia.

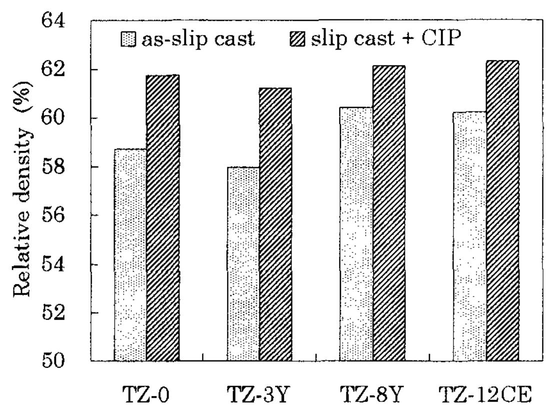

Fig. 2 Effect of the kind of zirconia particles on the relative densities of the specimens compacted by slip casting and followed by cold isostatic pressing at 400MPa.

The relative densities of the specimens sintered at 1673K are shown in Fig. 3. The relative green density of the TZ-8Y dispersed compact is highest, but density of the sintered body is lowest. The densities of the sintered bodies containing TZ-3Y and TZ-12CE, which are tetragonal, are similar.

Figure 4 illustrates the microstructures of the sintered bodies. Alumina particles appear dark in contrast to the bright zirconia particles. Particle dispersion indicated by the rheology measurements represent the suspension as a whole but several partial agglomerations of zirconia particles are observed in the micrographs of the TZ-0 and TZ-12CE dispersed materials (Fig. 4(a) and (b)). In the large agglomerated areas, grain growth of zirconia particles has occurred and large pores are observed. In the TZ-3Y and TZ-8Y dispersed materials (Fig. 4(a) and (b)), zirconia particles are dispersed finely and homogeneously. The average zirconia particle size of the TZ-3Y dispersed material is smaller than that of the TZ-8Y dispersed material.

The average alumina particle sizes in the materials containing TZ-0, TZ-3Y, TZ-8Y, and TZ-12CE were 0.44, 0.40, 0.42, and 0.50 μm, respectively. The alumina particle size of the TZ-

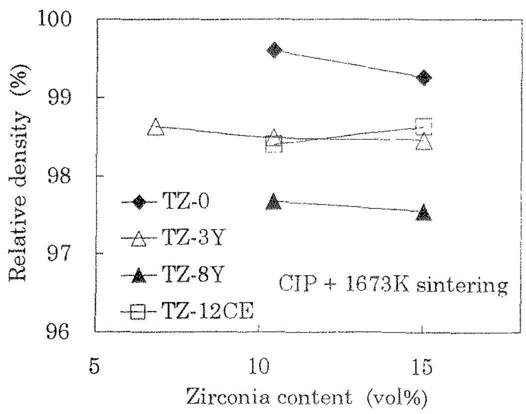

Fig. 3 Effect of zirconia content on relative densities of the various zirconia dispersed alumina sintered at 1673K.

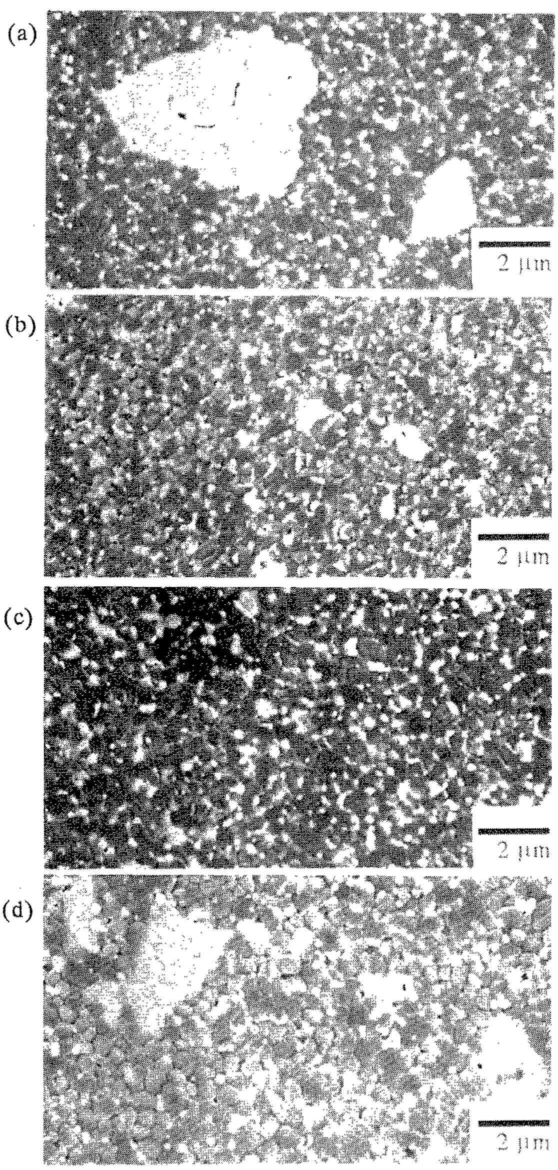

Fig. 4 Microstructures of sintered specimens. (a) is the 15vol% TZ-0 dispersed alumina, (b) is the 15vol% TZ-3Y dispersed alumina, (c) is the 15vol% TZ-8Y dispersed alumina, (d) is the 15vol% TZ-12CE dispersed alumina.

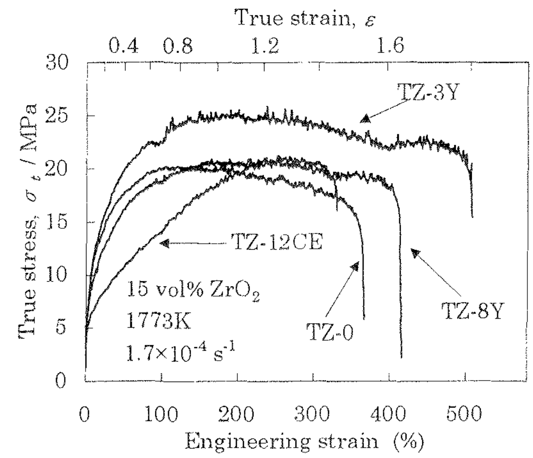

Fig. 5 Stress-strain curves of the 15vol% various zirconia dispersed alumina produced by colloidal processing. Tensile tests were conducted at 1773K using an initial strain rate of $1.7 \times 10^{-4} s^{-1}$.

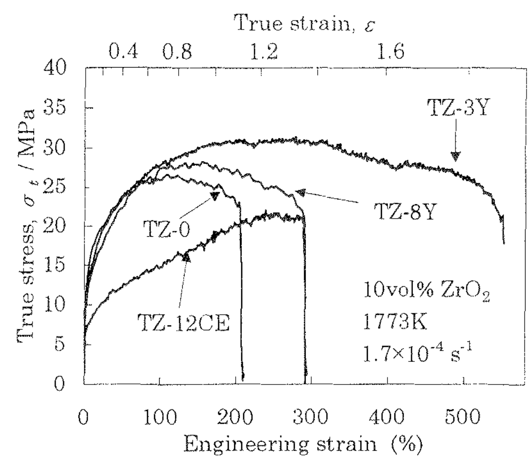

Fig. 6 Stress-strain curves of the 10vol% various zirconia dispersed alumina produced by colloidal processing. Tensile tests were conducted at 1773K using an initial strain rate of $1.7 \times 10^{-4} s^{-1}$.

12CE dispersed material was the largest because of reduced zirconia pinning, as a result of particle agglomeration. The average alumina particle size of the TZ-3Y dispersed material was smaller than that of the TZ-8Y dispersed material. That seems to be caused by the smaller zirconia particles in the TZ-3Y dispersed material. The relative densities of the zirconia dispersed materials were more than 98% theoretical density after sintering at a lower temperature as shown in Fig. 3. The fine microstructure with alumina grain size less than 0.5 μm is caused by the fine and homogeneous dispersion of zirconia particles and lower sintering temperature than conventional proc-essing.

True stress-strain curves of 15 vol% zirconia dispersed alumina for the tensile tests at 1773K are shown in Fig. 5. The total elongation of all specimens was observed to be more than 300%, and they exhibit superplasticity. Large elongation was possible due to the dense and fine

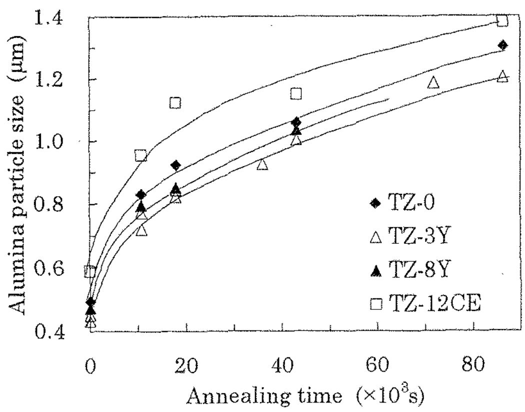

Fig. 7 Effects of various zirconia on alumina grain growth of the 10 vol% zirconia dispersed alumina produced by colloidal processing.

microstructure produced by colloidal processing. The total elongation of the TZ-3Y dispersed material exceeded 500%. The maximum elongation of the TZ-8Y dispersed material was inferior to the TZ-3Y dispersed material. The microstructure of the TZ-8Y was well dispersed, but it had a larger alumina particle size and lower density than TZ-3Y. The strain hardening of the TZ-12CE dispersed material remarkably appears for a wide range of strain. Strain softening begins at a smaller tensile strain in the TZ-0 dispersed material than in the TZ-8Y dispersed material. This difference in strain softening suggests that cavities accumulate in the TZ-0 dispersed material faster than in the material containing TZ-8Y.

Figure 6 shows tensile flow curves of the 10 vol% zirconia dispersed alumina materials. The total elongation of the material containing TZ-3Y was more than 500%, and the properties of strain softening and strain hardening were similar to the 15 vol% zirconia dispersed alumina materials.

Figure 7 represents the alumina grain size as a function of annealing time at 1773K. The alumina grain growth rate in the TZ-3Y dispersed material was the slowest. The largest elongation is attributed to initially small grain size and the suppression of alumina grain growth due to zirconia pinning. Fine and homogeneous TZ-3Y dispersion is very effective in preventing alumina grain growth. The grain growth rate in the TZ-12 dispersed material was fastest for all the materials tested.

Alumina grain growth measurements suggest that the differences in total elongation were caused by alumina grain growth during deformation. The phenomenon of strain hardening in the TZ-12CE dispersed material was caused by rapid alumina grain growth as a result of a suppression of the zirconia pinning mechanism.

4. SUMMARY

The differences in the kind of zirconia used for dispersion, and the distribution of the zirconia particles both have an influence on the microstructure and the tensile ductility of the zirconia dispersed alumina. TZ-3Y particles disperse finely and homogeneously in alumina and are most effective in improving the microstructure and tensile ductility due to a suppression of alumina grain growth.

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