

IMPROVEMENT OF MECHANICAL PROPERTIES OF ALUMINA PARTICULATE COMPOSITES USING POLYCRYSTALLINE ALUMINA POWDER

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ABSTRACT

Alumina particulate zirconia and alumina particulate glass using polycrystalline alumina powder was fabricated by atmospheric sintering to investigate the influence of alumina grain distribution on the mechanical properties of alumina composites. Polycrystalline alumina powder was obtained by quenching and crushing sintered alumina body. Firstly, the fracture toughness of 20% of zirconia particulate alumina was slightly increased with the addition of polycrystalline alumina powder. On the other hand, the bending strength was kept almost constant with the addition of polycrystalline alumina powder. SEM observation of a crack path indicated that a crack propagated into alumina cluster formed by polycrystalline alumina powder and transgranular fracture often occurred in the cluster compared to the other region. Second, 50% of alumina particulate borosilicate glass was sintered using virgin alumina powder or polycrystalline alumina powder. Using polycrystalline alumina powder, sintered body was obtained at lower sintering temperature than that using virgin alumina powder and the average bending strength was improved by the addition of polycrystalline alumina powder, too. Some samples showed that mullite formation occurred during sintering and X-ray analysis indicated that the addition of polycrystalline alumina powder enhanced mullite formation in the samples. These results indicate that the addition of polycrystalline alumina powder can improve fracture toughness, bending strength, mullite formation and/or low temperature sintering of alumina particulate composites.

KEYWORDS

composite, alumina, zirconia, glass and mechanical properties.

INTRODUCTION

Ceramics or glass matrix composites have been studied to improve the poor mechanical reliability of ceramics and glass for the practical use in various industries [1-3]. Conventionally, microstructure of ceramics has been

designed and controlled on a singular scale level. Natural materials (e.g. bamboo), however, consist of complicated structural elements on the several scale level and achieve the excellent combinations of mechanical properties (e.g. flexibility, toughness and stiffness). In the recent studies, synergy ceramics has been proposed and some artificial hyper-organized structure controlling ceramics were fabricated [4]. Nano ceramics composites, for example, exhibited better fracture strength than conventional ceramics [5,6] and the fracture toughness of Si_3N_4 was enhanced by elongated coarse grains [7].

The idea of nano-composites and coarse grain bridging was based on the mixture of dual scale particles. On the other hand, the mixture of dual scale microstructures is also attractive for design of synergy ceramics. Simple example is the composites where clusters of ceramics A are dispersed in matrix of ceramics B. In this case, if the cluster bridging occurs, the toughening by the cluster bridging would be more effective than that by the grain bridging in the conventional ceramics composites because cluster size is much larger than fine singular grain. Though the coarse singular gains dispersed composites can be expected to get the same effect of the toughening by cluster bridging, it usually shows significant decrease in fracture strength caused by inter/trans granular fracture along/in coarse grains. Polycrystalline alumina clusters dispersed composites, however, could maintain the same fracture strength of conventional fine ceramics because the clusters consist of fine singular grains. In this study, alumina polycrystalline powder was obtained by crushing pre-sintered alumina body, mixed with alumina-zirconia composites or Pylex glass. Polycrystalline clusters dispersed composites were obtained by atmospheric sintering and measurements of mechanical properties, observation of microstructure and phase detection by XRD were performed.

EXPERIMENTAL PROCEDURE

Commercially available alumina powder (Sumitomo Chemical Co., Ltd., AES-11) was used for polycrystalline powder preparation. Alumina powder was pressed by cold isostatic press under 200 MPa and sintered in air at 1873K for 2h. Sintered alumina body was crashed by a stamp-mill and a automated mortar and sieved through the mesh of which opening size is $32\mu\text{m}$. Obtained polycrystalline alumina powder was mixed with virgin alumina powder and commercially available zirconia powder (Tosho Co., Ltd., TZ-3Y) by a wet ball-mill process in ethanol for 24h and dried by a rotary evaporator. The dried powder was pressed and sintered under the same condition mentioned above. The volume fraction of polycrystalline alumina, virgin alumina and virgin zirconia powder was shown in Table1. The volume fraction of total amount of alumina powder (virgin and polycrystalline power) and zirconia powder was set to 80 vol% : 20 vol%. Commercially available Pylex glass powder (Furuya Metal Co., Ltd., 80.9 mol% SiO_2 , 12.7 mol% B_2O_3 and 2.3 mol% Al_2O_3) was used for processing of alumina particulate glass matrix composites. The average diameter of Pylex glass powder was $2\mu\text{m}$. Polycrystalline or virgin alumina powder was mixed with Pylex glass powder, dried and pressed and under the same condition mentioned above. The pressed body was sintered in air for 9h. Volume fraction and sintering temperature were shown in Table2. Obtained sintered body was cut to the specimen of 3 x 4 x 40mm and four-point bending test was done. The bending test was performed using a universal testing machine with a cross-head speed of 0.5 mm/min, an upper span of 10 mm and a lower span of 30mm. The microstructure of polished and etched surface of specimens was observed a scanning electron microscope (SEM). Specimens of alumina-zirconia composites were also served for fracture toughness measurement. Vicker's indenter was loaded on the specimen surface under 30kgf for 30sec to induce a crack perpendicular to long axis of a specimen. The four-point bending test was performed to the indented specimens, which were loaded tensile stress on indented surface and the fracture strength was measured. The length and depth of the indented crack on fracture surface was measured by optical microscope and the fracture toughness was calculated using Newman's equation [8]. The path of indented crack on the surface was also observed by SEM. The phases in specimens of alumina-glass composites were identified by X-ray diffraction analysis (XRD) using $\text{Cu K}\alpha$ radiation.

TABLE 1
COMPOSITION OF ALUMINA-ZIRCONIA SAMPLES

Sample	Virgin Alumina Powder (vol%)	Polycrystalline Alumina Powder (vol%)	Virgin Zirconia Powder (vol%)
AZ0	80	0	20
AZ1	70	10	20
AZ2	60	20	20
AZ3	50	30	20

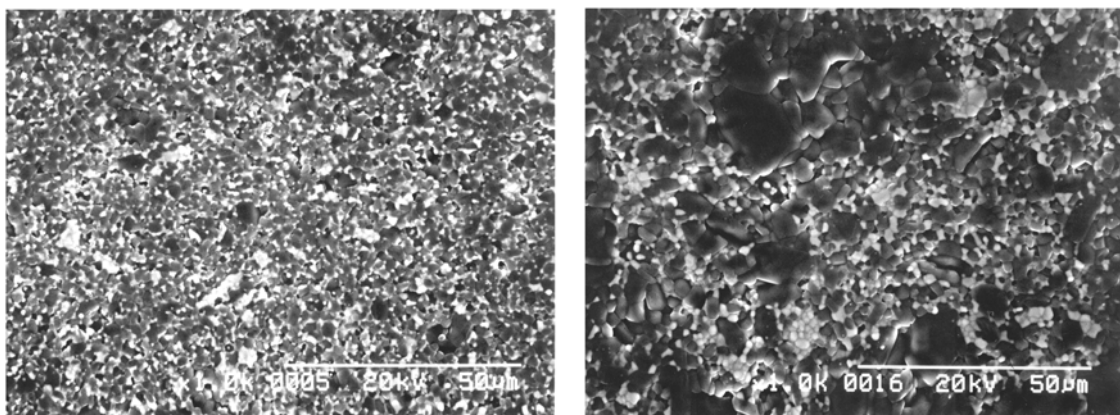
TABLE 2
COMPOSITION OF ALUMINA-GLASS SAMPLES

Sample	Virgin Alumina Powder (vol%)	Polycrystalline Alumina Powder (vol%)	Pylex Glass Powder (vol%)	Sintering Temperature (K)
GV1	50	0	50	1273-1573
GP1	0	50	50	1173-1573

RESULTS AND DISCUSSION

Alumina Particulate Zirconia Matrix Composites (Alumina-zirconia)

Figure 1 shows the microstructure of alumina-zirconia samples. The samples using only virgin powder shows that fine grains of alumina and zirconia were homogeneously distributed. The samples with the addition of polycrystalline powder show that polycrystalline alumina clusters were dispersed in fine alumina-zirconia matrix and dual scale microstructure was obtained. The coarsening of alumina grains, however, was observed in polycrystalline alumina clusters. Polycrystalline alumina powder was obtained by pre-sintering and thus polycrystalline alumina clusters were suffered double sintering. Such a repeat of sintering caused the coarsening of alumina grains and the further consideration of sintering condition should be needed to obtain fine sintered body. Figure 2 shows the bending strength of alumina-zirconia specimens. The bending strength was slightly increased with the addition up to 20 vol% of polycrystalline alumina powder and dropped with the addition of 30 vol% of that. The decrease in bending strength with the addition of 30 vol% of



(a)

(b)

Figure 1: Microstructure of the thermal etched surface of 20 vol% of zirconia particulate alumina. (a) the sample using only virgin alumina powder (b) the sample adding 20 vol% of polycrystalline alumina powder.

polycrystalline alumina powder could be related to the coarsening of polycrystalline alumina clusters due to double sintering mentioned above. On the other hand, the addition up to 20 mol% of polycrystalline alumina powder enhanced or kept constant of the bending strength of the samples regardless of coarsening of polycrystalline alumina clusters. This result indicates that the dual structure formed in alumina-zirconia samples may contribute on the improvement of bending strength.

Figure 3 shows the fracture toughness of alumina-zirconia samples. Fracture toughness was apparently increased with the addition of polycrystalline alumina powder. Figure 4 shows a crack path of samples induced by Vicker's indentation. In the case of the samples using only virgin powder, intergranular fracture largely dominated a crack path. In the case of the sample adding polycrystalline alumina powder, however, both of intergranular and transgranular fracture were observed in the cluster region of polycrystalline alumina grains and intergranular fracture largely dominated in the other region. It is noted that a crack deflection along polycrystalline alumina clusters had never been observed. This fact points out that the cluster bridging by polycrystalline alumina grains unfortunately did not occur in any alumina-zirconia samples although the grain bridging by alumina and zirconia particles were often observed in the mixture region of alumina and zirconia. Possible reason why the fracture toughness was enhanced by the addition of polycrystalline alumina powder is the change of the internal stress distribution in composites. The thermal expansion coefficient of zirconia is higher than that of alumina and consequently the tensile stress occur in the zirconia particles due to thermal residual stress in the case of the samples where zirconia particles are uniformly distributed in the alumina

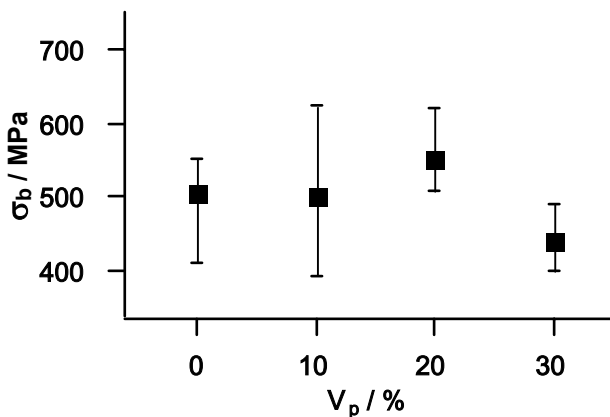


Figure 2: Relationship between bending strength (σ_b) and volume fraction (V_p) of polycrystalline alumina powder.

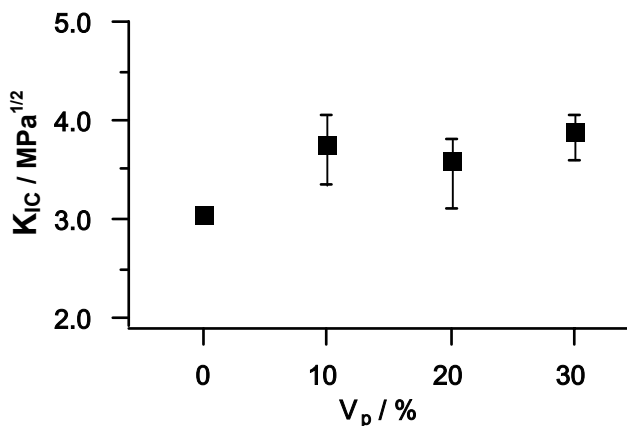


Figure 3: Relationship between fracture toughness (K_{IC}) and volume fraction (V_p) of polycrystalline alumina powder.

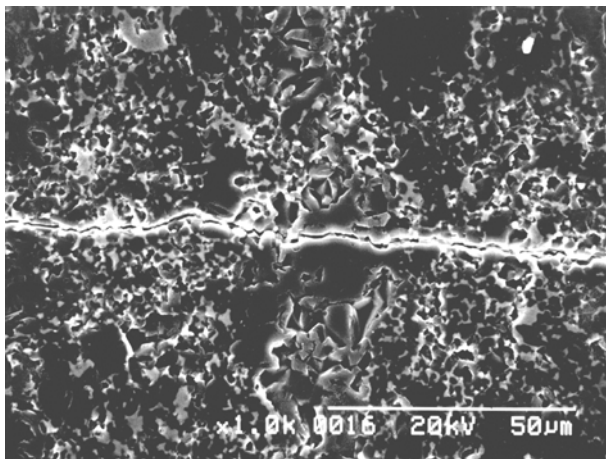


Figure 4: Indented crack path on the etched surface of the sample adding 20 vol% of polycrystalline alumina powder.

matrix. On the other hand, the mixture region of alumina and zirconia has higher macroscopic thermal expansion coefficient than the cluster region of polycrystalline alumina and thus the compressive stress occur in the polycrystalline alumina cluster in the samples where polycrystalline alumina clusters are distributed in the mixture structure of alumina and zirconia. SEM observation indicates that a crack propagated into the polycrystalline alumina cluster. If the compressive stress prevents crack propagation in the polycrystalline alumina cluster, the fracture toughness would be enhanced. Of course, further study is needed because grain size distribution, crack deflection, crack bowing and the other factors should be considered to explain the toughening mechanism by the addition of polycrystalline alumina powder.

Alumina Particulate Glass Matrix Composites (Alumina-glass)

Figure 5 shows typical XRD pattern of the alumina-glass samples. The samples using virgin alumina powder shows that the formation of mullite occurred when sintering temperature was higher than 1473K. On the other hand, the samples using polycrystalline alumina powder shows the mullite formation occurred when sintering temperature was higher than 1373K. The peak ratio of mullite (peak B) to alumina (peak A) exhibits 0.25 for the samples using virgin alumina and 0.46 for the samples using polycrystalline alumina powder when sintering temperature was 1473K. The addition of polycrystalline alumina powder, therefore, enhances mullite formation. Possible reason of enhancement of mullite formation is impurities mixed at preparation of polycrystalline powder. Figure 6 shows microstructure of the samples with mullite formation. Fibrous mullite

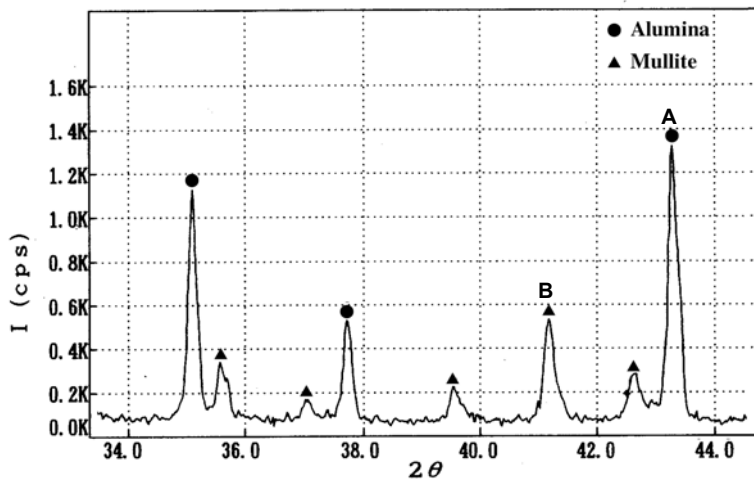


Figure 5: XRD pattern of alumina particulate Pylex glass using polycrystalline alumina powder (sintering temperature is 1473K).

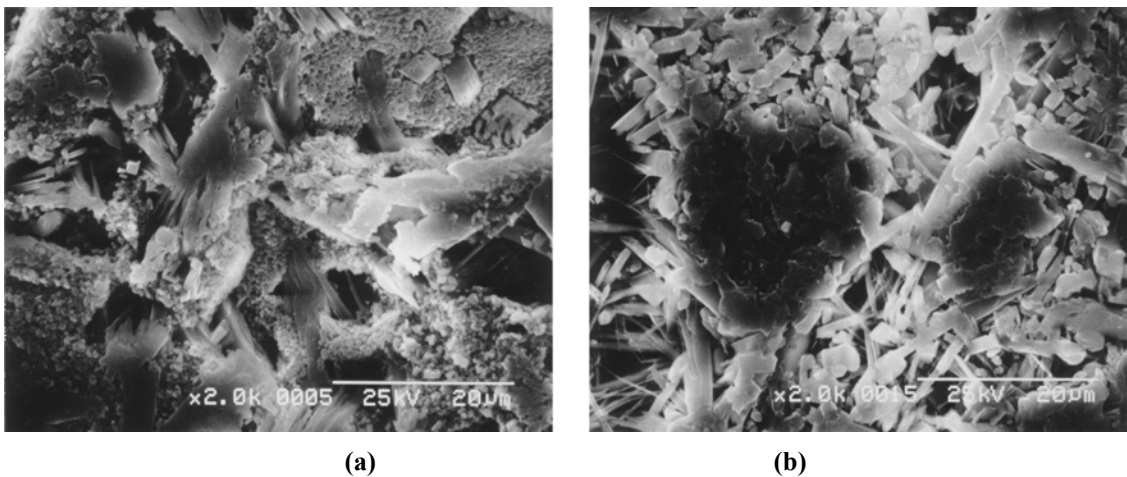


Figure 6: Microstructure of the etched surface of alumina particulate Pylex glass sintered at 1473K. (a) the sample using virgin alumina powders (b) the sample using polycrystalline alumina powder.

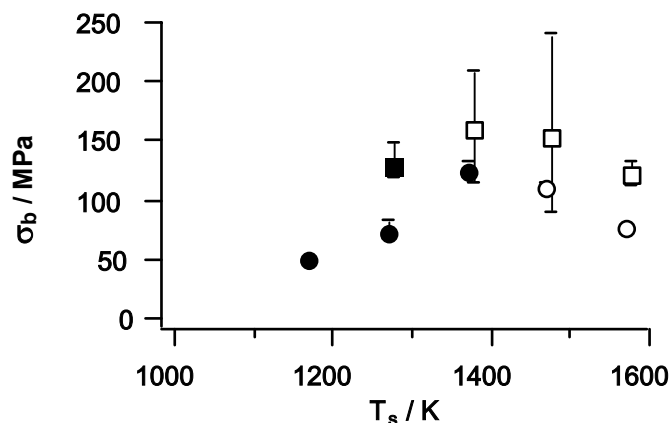


Figure 7: Relationship between bending strength and sintering temperature of alumina particulate Pylex glass. Opened marks show that mullite formation occurred.

crystals were largely formed and polycrystalline alumina clusters still remained in the mixture of glass, alumina and fibrous mullite in the samples, which shows the interested dual structure. Figure 7 shows relationship between the bending strength and sintering temperature of alumina-glass samples. The bending strength was apparently enhanced by the use of polycrystalline alumina powder. Mullite formation usually contributes on the strengthening of glass ceramics but, in this case, the samples with mullite formation tended to have larger scatter of bending strength than those without mullite formation.

CONCLUSIONS

Alumina particulate zirconia and alumina particulate glass using polycrystalline alumina powder was fabricated by atmospheric sintering and composites with dual scale structures were obtained.

The samples of dual scale structures where polycrystalline alumina clusters were distributed in the mixture of alumina and zirconia were obtained though polycrystalline alumina clusters showed coarsening by double sintering. These samples indicated that the addition of polycrystalline alumina powder enhanced the fracture toughness while the bending strength was almost kept constant.

The samples of dual scale structures where polycrystalline alumina clusters were distributed in the glass matrix were obtained. When mullite formation occurred, the polycrystalline alumina clusters were distributed in the mixture of glass and fibrous mullite. The use of polycrystalline alumina powder enhanced the mullite formation and the average bending strength though mullite formation causes the increase in scatter of the bending strength.

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