Journal of Ceramic Processing Research. Vol. 14, No. 2, pp. 210~212 (2013)

J O U R N A L O F
Ceramic
Processing Research

Effect of SiO_2, $CaCO_3$ and talc on sintering behavior of ZTA

Jihoon Chae and Bumrae Cho
Department of Materials Engineering, Keimyung University, Daegu 704-701, Korea

Sintered zirconia toughened alumina (ZTA) which has good mechanical properties at a low temperature was produced by milling and mixing with Al_2O_3 and ZrO_2. In order to examine the effect of talc, SiO_2 and $CaCO_3$ on mechanical properties of ZTA, microstructure and density change were observed. The addition of sintering aid induced densification of ZTA at low temperatures. This inducement of densification is believed as the result of addition of the sintering aids which lowered the sintering temperature and generated rapid mass transfer through liquid phase. In result, as the amount of sintering aids and sintering temperature increased, the densification was suppressed, and the formation of pores showed a tendency to be increased. This result is known as due to the vaporization of liquefied sintering aids.

Key words: ZTA, Sintering aids, Phase transformation.

Introduction

Among many ceramic materials, Alumina (Al_2O_3) has high levels of thermal resistance, corrosion resistance and mechanical strength, enabling it to be used in a variety of products, such as abrasion materials, spark plugs, ceramic tiles and organic materials [1]. In order to overcome the brittle fracture phenomenon of the ceramic composites, there are several methods such as deflecting the crack propagation path by utilizing the residual stress induced due to different thermal expansion coefficient between the matrix and the secondary phase, increasing the fracture toughness by inducing needle-type particles or whiskers that have a high aspect ratio, into the ceramic matrix, increasing the crack propagation energy [2-4]. Many researchers have focused on the densification of ceramics, and it is revealed that when conducting solid state sintering, the sintering temperature is lowered by adding much finer powder that has a better sintering ability, or by inducing the liquid phase sintering during the process. Claussen et al. have reported that the improvement of fracture toughness of ZrO_2 induced Al_2O_3 matrix, known as zirconia toughened alumina (ZTA), differs from partially stabilized zirconia (PSZ) in that it is explained by multiple mechanisms [5-7].

In this research, the effects of SiO_2, $CaCO_3$, and talc as the sintering aids on the microstructural changes of ZTA were examined to produce the ZTA having good mechanical properties at low temperatures. The relationship between the densification behaviors of ZTA and those sintering aids was also investigated.

*Corresponding author:
Tel : +82-53-580-5359
Fax: +82-53-580-5359
E-mail: chobr@kmu.ac.kr

Experimental

Average particle size of Alumina (Al_2O_3, Sumitomo Co. Ltd., Japan) used in this research was 0.69 μm and 20 ~ 30 wt% of Zirconia (3Y-TZP, Terio Co. Ltd., China) that had a average particle size of 0.12 μm was added to change the composition of ZTA. For the sintering aids, talc ($Mg_3(OH)_2$ $(Si_2O_5)_2$), KOCH Co. Ltd., Korea), $CaCO_3$ (Dongho calcium Co. Ltd., Korea), and SiO_2 (Nycontech Co. Ltd., Korea) were used. After adding 5 wt% of the sintering aid in ZTA powder, the materials were crushed and mixed by using a attrition mill. The produced slurry was analyzed by a particle size analyzer (LS-230, Coulter, USA) and had a average particle size of 0.35 μm. The completely crushed and mixed slurry was then dried in a microwave oven. After drying, each sample was grinded and sieved under 75 μm for forming. After the sieving process, the materials were formed by a uniaxial compression tester and a metal mold under 30 MPa pressure. The formed specimens were sintered for 3hours under 1300 °C ~ 1500 °C temperature by a electrical furnace and were furnace cooled. Apparent density of each sample was measured by a gravis-meter (MC1, Sartorius, Japan) with the Archimedes' principle. The hardness of the materials was measured by the Micro Vickers hardness tester (MX7164 Alpha, Matusuzawa Co., Japan). In order to observe morphology of particles and degree of densification of the specimens obtained at different sintering temperatures, a scanning electron microscopy (S4200, Hitachi Co., Japan) was used after proper thermal etching.

Results and Discussion

The specimens that were sintered at temperatures over 1450 °C showed consistency of more than 98% as

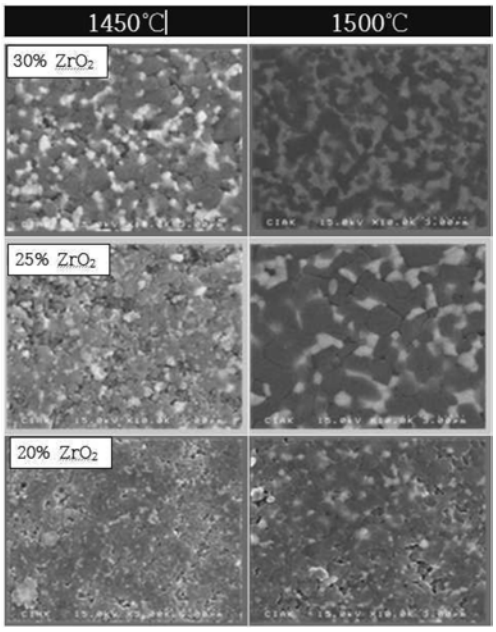

Fig. 1. SEM micrographs of ZTA specimens containing different contents of ZrO_2 sintered at 1450 °C and 1500 °C without any sintering aids.

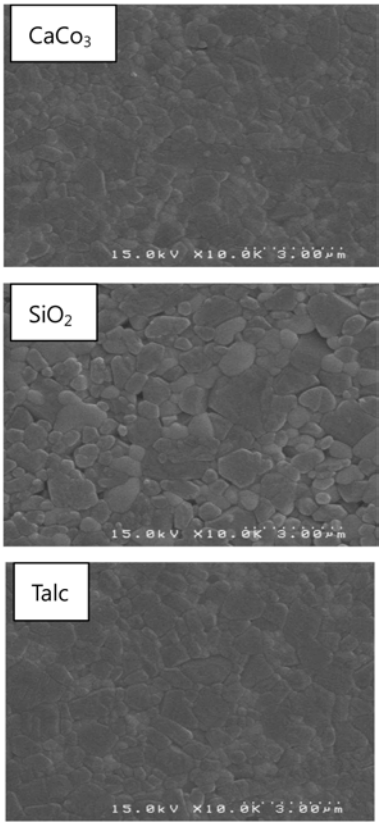

Fig. 2. SEM micrographs of ZTA specimens sintered at 1450 °C with different kinds of sintering aids.

compared to the theoretical density. In the case of the specimens without sintering aids, when 3Y-TZP was mixed, the resultant micro structure of specimens showed nearly uniform size distributed grains indicating

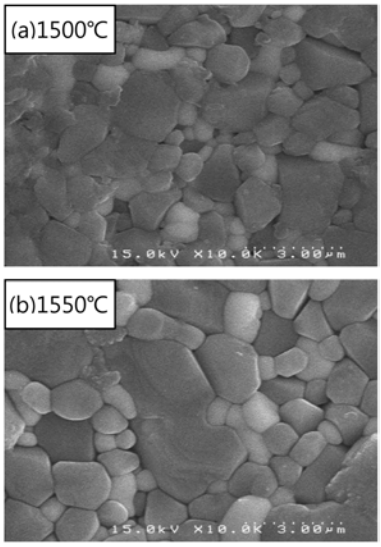

Fig. 3. SEM micrographs of ZTA specimens containing talc sintered at (a) 1500 °C and (b) 1550 °C.

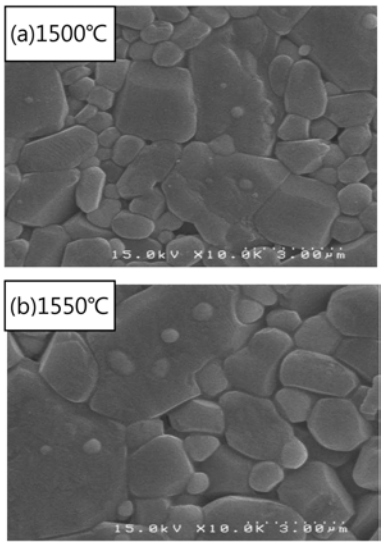

Fig. 4. SEM micrographs of ZTA specimens containing SiO_2 sintered at (a) 1500 °C and (b) 1550 °C.

that the grain growth of Al_2O_3 was suppressed. Furthermore as the amount of ZrO_2 was increased, the grain growth of ZrO_2 was also restrained leading to the reciprocal suppression as Al_2O_3 and ZrO_2 combine. This is thought to be due to the formation of Al_2O_3 and ZrO_2 ternary system with Y_2O_3 as Y_2O_3 added therefore raising the solubility level of ZrO_2 and Y_2O_3.

Fig. 1 shows SEM micrographs of ZTA specimens containing 20 wt%, 25 wt% and 30 wt% of ZrO_2 sintered at 1450 °C and 1500 °C without any sintering aids. From the micrographs, the specimen containing 20 wt% of ZrO_2 had many pores regardless of the sintering temperature. However, the specimens containing over 25 wt% of ZrO_2 showed a dense microstructure and it is believed that the reciprocal suppression had occurred between Al_2O_3 and ZrO_2.

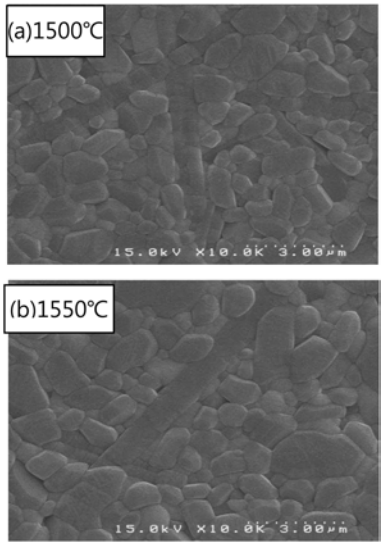

Fig. 5. SEM micrographs of ZTA specimens containing $CaCO_3$ sintered at (a) 1500 ℃ and (b) 1550 ℃.

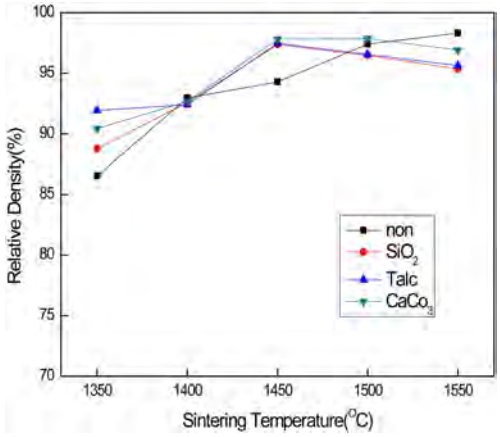

Fig. 6. Relative density of ZTA specimens sintered at various temperatures.

Fig. 2 shows SEM micrographs of the specimens containing $CaCO_3$, SiO_2 or talc as the sintering aid sintered at 1450 ℃ for 3 hours. From the experimental result that many pores were observed in all micrographs, it is clear that the effect of the sintering aids on densification behavior was insignificant at this temperature. Liquefaction trace due to sintering aids reacting was observed between Al_2O_3 grains.

The SEM micrographs of Fig. 3 shows the microstructure of the specimens containing talc sintered at different temperatures. The specimens sintered at 1500 ℃ showed nearly uniform and dense distribution of fine grains, while specimens sintered at 1550 ℃ showed abnormal grain growth and micro pores between grains due to evaporation of talc.

Similar microstructure was observed in the specimens containing SiO_2 as shown in Fig. 4. Fig. 4(a) shows the microstructure of the specimen sintered at 1500 ℃. At this temperature, it was observed that SiO_2 already had begun to evaporate, resulting in considerable pore formations along with large particles. In the case of specimens sintered at 1550 ℃, abnormal particle growth, leading to larger pores were observed as shown in Fig. 4(b).

Fig. 5 shows the SEM micrographs of the specimens containing $CaCO_3$ sintered at 1500 ℃ and 1550 ℃. Unlike in Figs. 3 and 4, long and narrow grains which were elongated to a particular direction were observed in Fig. 5. The elongated grains may effect on improving the fracture toughness. Also at the temperature of 1550 ℃, compared to other specimens including SiO_2 or talc, the abnormal grain growth seems to be suppressed more, resulting in higher densification.

Fig. 6 shows the relative density of each specimen. As seen, specimens with uniform distribution of fine grains shows higher relative density compared to specimens with abnormal grain growth. This can be explained by formation of liquefied phases when the sintering aids reacted with alumina during the process, leading to liquid sintering [8].

Conclusions

This study has examined the effects of sintering aids on microstructural change of ZTA. Specimens without sintering aid showed uniform grain size distribution as compared to those with sintering aid that showed abnormal grain growth due to formation of liquefied phases. Especially, the specimens containing $CaCO_3$ had the most elongated grains. As sintering temperature increases, larger and abnormal grain growth were observable. The specimens that had abnormal grains also showed larger pore formation, leading to negative effect on densification behaviors of ZTA. Densification is suppressed as the amount of sintering aids and sintering temperature increase. The density measured at low temperatures showed higher in the order of Talc < $CaCO_3$ < SiO_2.

References

1. W. Dwork and Fingerle, J. Brit. Ceram. Soc. 86 (1987) 170-178.
2. C. Greskovich and J.A. Palm, J. Am. Ceram. Soc. 639 (1980) 597-599.
3. R. Lundberg, L. Kahman, R. Pompe, R. Carlsson and R. warren, Am. Ceram. Soc. Bull. 67 (1987) 333-338.
4. B. Cho, J. Chae, B. Kim and J. Kang, Matls. Sci. Foru, 724 (2012) 249-254.
5. N. Claussen, J. Am. Cerm. Soc. 59 (1976) 49-51.
6. R.M. McMeeking and A.G. Evans, J. Am. Ceram. Soc. 65 (1982) 242-246.
7. K.T. Faber and A.G. Evans, Acta Metall. 31 (1983) 565-576.
8. W.D. Kingery and M.D. Narasimhan, J. Appl. Phys. 30 (1959) 307-310.