Compaction and Sintering Behavior of Al_2O_3-modified Ziroconium Titanate ($ZrTiO_4$)

Myoung Pyo Chun[1,a], Hur Geun[1,b], Seung Jae Myoung[1,c], Jung Ho Cho[1,d], and Byung Ik Kim[1,e]

[1]Advanced Materials & Components Team, Korea Institute of Ceramic Eng. & Tech.,
233-5, Gasan-Dong, Guemcheon-Gu, Seoul, 153-801, Korea
[a]myoungpyo@kicet.re.kr, [b]gjrms3@nate.com, [c]msj7179@hanmail.net,
[d]goedc@kicet.re.kr, [e]bikim@kicet.re.kr

Abstract

The compaction and sintering behavior of zirconium titanate ($ZrTiO_4$) was investigated by means of the measurement of sintering density and shrinkage, and the observation of microstructure. With increasing the content of Al_2O_3 additive, Al2O3-modified zirconium titanate samples fired at 1300°C showed the anisotropic shrinkage behavior that the upper region of sintered body has higher sintering shrinkage than the low region. This difference of sintering shrinkage decreased with increasing firing temperature from 1300 to 1400°C. The SEM micrographs of powder compation show that the anisotropic shrinkage behavior is related with non-uniform density in a uniaxial compaction.

Keywords : Zirconium titanate, compaction, sintering, shrinkage, density

1. Introduction

Zirconium titante (TiZrO4) has been utilized as dielectric resonators and it is also interesting for a wide range of applications including catalyst and high temperature pigment, etc.

Many advanced engineering ceramics usually require small dimensional tolerances. The green compact may show deviations from the original die geometry after firing. Therefore, the controlling of sintering shrinkage is very important for a geometrically scaled replica of the corresponding unfired green body.

In this study, the compaction and sintering behavior of Al_2O_3 modified $ZrTiO_4$ (ZT) were investigated by means of the measurement of sintering density and shrinkage, and the observation of microstructure.

2. Experimental and Results

Starting materials were powders of ZrO_2, TiO_2, and Al_2O_3. $ZrTiO_4$ (ZT) powder was synthesized by the solid state reaction of thoroughly ground mixtures of ZrO_2 and TiO_2 powders that were milled in the required stoichometric ratio. The milling operation was carried out for 6 h in ethanol using ZrO_2 balls. After drying at 60°C in vacuum oven, the mixed powder was calcined at various temperatures ranging from 1130 to 1240°C in order to synthesize the zirconium titanate. Calcined powders were investigated by X-ray diffraction to identify the phases formed as shown in Fig.1. It should be noted that $ZrTiO_4$ (ZT) of orthorhombic structure is formed at 1240°C.

To study the sintering behavior of ZT as a function of the content of Al2O3, the compactions with compositions of (100-*x*)ZT - (*x*)Al2O3 (*x* = 0, 5, 10wt%) were prepared by ball milling for 1 h, binder mixing (weight % ratio; powder/PVA =85/15) for 12 h, and uniaxial pressing to produce pellets 40 mm in length ×30 mm in width ×10 mm in height. Pellets were sintered at temperatures between 1300 and 1500°C for 3 h in air.

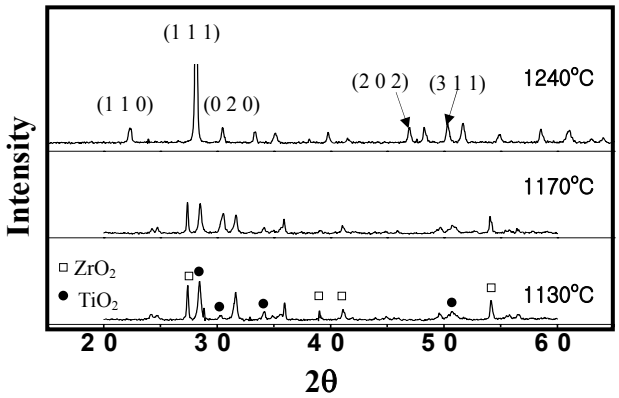


Fig. 1. XRD patterns of ZT powder calcined at three temperature.

Fig.2 shows the ceramic samples sintered at 1300, 1400, and 1500°C. For the samples sintered at 1300 °C, it should be noticed that camber appears with increasing the content of Al_2O_3 in ZT. However, these cambers disappear with increasing sintering temperature above1400 °C.

The ceramic samples are very porous in sintering at

1300 °C and dense in sintering at 1500 °C as shown in Fig.3. Therefore, the camber formed in the samples sintered at 1300 °C seems to be related with the difference of green density or sintering shrinkage between upper and lower region of pellets.

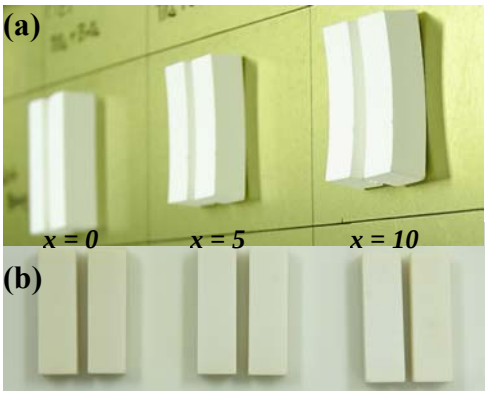
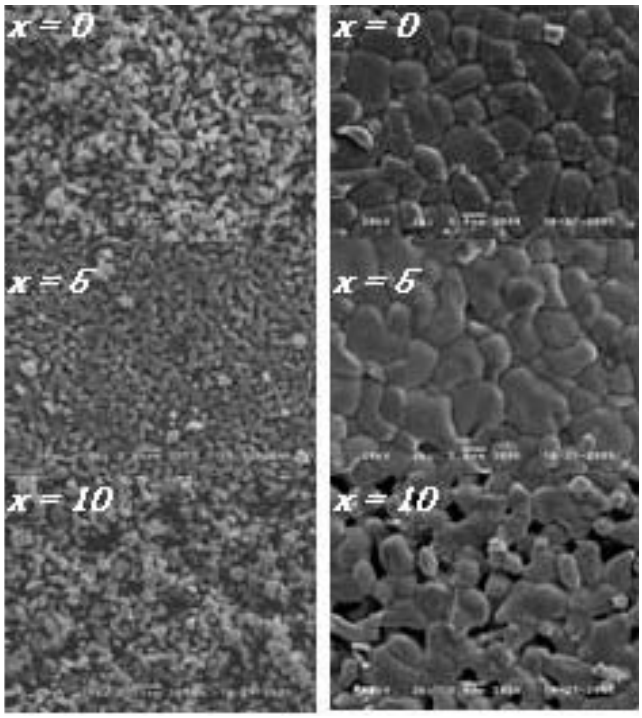


Fig. 2. Photographs of samples sintered at (a) 1300oC, and (b) 1400oC for the compositions of (100-*x*) TZ - (*x*)Al2O3 (*x* = 0, 5, 10 wt%).



(a) (b)

Fig. 3. SEM micrographs of samples sintered at (a) 1300°C, and (b) 1500°C for the compositions of (100-*x*)TZ - (*x*)Al2O3 (*x* = 0, 5, 10 wt%).

Fig.4. shows the SEM micrographs of the uniaxially pressed compact of composition (95wt%TZ – 5wt%Al2O3). It is shown that some cracks are observed in the lower region of green compact. So, the green density of the lower region seems to be less dense than that of the upper region. The camber that appears for the samples sintered at 1300°C is considered to originate from the difference of green density between upper and lower region of compact.

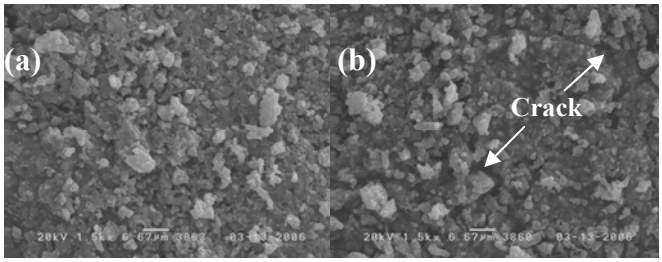


Fig. 4. SEM micrographs of compact with composition of 95wt%TZ–5wt%Al2O3 uniaxially pressed at 1 ton force. (a) upper region, and (b) lower region of green compact.

The reason that the camber disappears for the samples sintered above 1400°C seems to be related with mass transport of particles and diffusion of atoms toward less dense compact region during the sintering.

3. Summary

The compaction and sintering behavior of zirconium titanate ($ZrTiO_4$) was investigated by means of the measurement of sintering density and shrinkage, and the observation of microstructure. In case of Al2O3-modified zirconium titanate (ZT), camber appears for the samples sintered at 1300°C. But, this camber disappears with increasing the sintering temperature above 1400°C. The anisotropic shrinkage behavior is considered to be related with the difference of green density between the upper and lower region of green compact.

4. References

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