Effect of sintering process parameters on the properties of 3Y-PSZ ceramics

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Abstract: The effect of sintering process parameters on the properties of 3 mol% yttria partially stability zirconia (3Y-PSZ) ceramics has been investigated. The relative density of the sintered pellet rapidly increases from 70.5 to 93.6 % with rose temperature from 1473 to 1573 K. In addition, the relative density only slightly increases from 94.9 to 96.6 %, when rose sintered temperature from 1573 to 1773 K. This result shows that no significant influence on the densification behavior when sintering at 1573 to 1773 K for 2 h. The Vickers hardness and toughness also increase with the sintered temperature.

1. Introduction

Although various types of ZrO_2 ceramics are currently available [1], however, only the Y_2O_3-doped tetragonal zirconia polycrystals (3Y-TZP), MgO-doped partially stabilized zirconia (Mg-PSZ) and zirconia toughened alumina (ZTA) are used to date in dentistry [2]. In the ZTA systems, the stress-induced transformation capability of ZrO_2 is to combine it with an alumina matrix created a ZrO_2-toughened alumina [3]. Therefore, these materials are recently received interest as potential bioceramics [4]. Moreover, Piconi and Maccauro [5] are demonstrated that the Mg-PSZ has not been successful for possible biomedical applications because the presence of porosity, associated with a large grain size of about 30-60 µm for induce wear.

In the previous studies of 3Y-TZP, have various studies on the fatigue behavior [6,7], the higher susceptibility to low temperature, degradation and lower reliability [8], effect of shape indentation damage on the long term performance [8,9] and the effect of $CaO-P_2O_5$ glass added on the micro structure and mechanical of 3Y-TZP dental ceramics [9]. However, the effect of sintering process parameters on the properties of 3Y-PSZ ceramics has not been investigated in detail. In the present study, the effect of sintering process parameters on the properties 3Y-PSZ ceramics with been discussed in detail.

Experimental procedure

2.1 Materials processing

High purity commercial 3Y-PSZ powders (supplied by Tosoh Inc., Japan) were blended for 4 h in a laboratory ball mill containing high purity ZrO_2 sintered balls. The resulting powder mixtures after shifting through a 200 mesh sieve, were compacted at room temperature by uniaxial pressing at 176 MPa in a 30 mm diameter stainless steel die. These compacted pellets well sintered at 1473- 1773 K for 2 h respectively, in a $MoSi_2$ furnace with a heating rate of 10 K/min, and then cooled in the furnace.

2.2 Sample characterization

The relatively bulk density was measured by the Archimedes method. Crystalline phases were identified by X-ray diffraction (XRD) and nano beam diffraction pattern (NBDP). The XRD was performed with a Rigaku X-ray diffractometer with Cu K_α radiation and Ni filter at a scanning rate of 1°/min. A transmission electron microscopy (JEM 2100F, JEOL, Tokyo, Japan) was used to observed the microstructure and determine the crystal structure. Hardness was determined using a Vickers indentation hardness tester (INDENTEMET 1150 SERIES, RUEHLER, USA). In each sample, 10 indentations were measured, under a load of 2000 gf and a holding time of 30 s.

3. Result and discussion

The XRD pattern of the 3Y-PSZ pellects after sintering at 1773 K for 2 h is shown in Fig. 1, which reveal that only the phases of $t-ZrO_2$ and $m-ZrO_2$ were appear. In fact, the phase transition of $t-ZrO_2$ to $m-ZrO_2$ was occurred for pure ZrO_2 from high temperature cooling to room temperature. This process is a reversible thermal martensitic transformation accompanied about 3 - 4.5 % volume expansion. For stabilized the $t-ZrO_2$ in the sintering microstructure of sintered pellets, several dopants such as Y_2O_3, MgO, CeO_2 and CaO are added to the ZrO_2. This process enhances the toughness because the energy associated with crack propagation in diesipated both in the transformation of $t-ZrO_2$ to $m-ZrO_2$ and in the overcoming of the compression stress is attributed to the volume expansion [9].

When the 3Y-PSZ pellet samples were sintering at various temperatures for 2 and 6 h, the relative densities of the 3Y-PSZ pellets as a function of sintering temperature are shown in Fig. 2. It is seen that the relative density of the sintered samples rapidly increases from 70.5 to 96.6 % when sintered at 1473 to 1773 K for 2 h. On the other hand, the relative density increases from 70.7 to 97.0 % when sintered at 1473 to 1773 K for 6 h. In this study, the sintering time prolong from 2 to 6 h has no significant influence on the densification behavior. Usually, the densification of ceramics is achieved by the shrinkage of the open pores and the grain boundary diffusion.
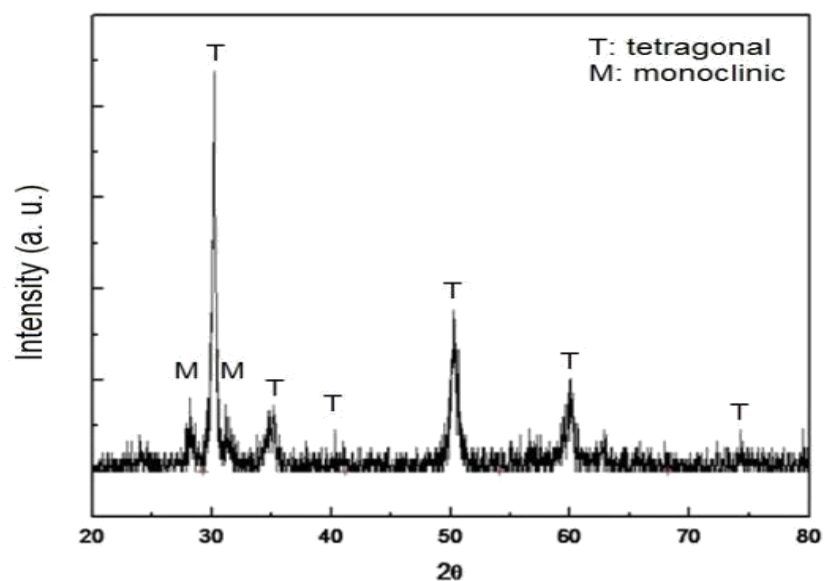
Figure 1. XRD pattern of the 3Y-PSZ pellet sample samples sintered at 1773 K for 2 h
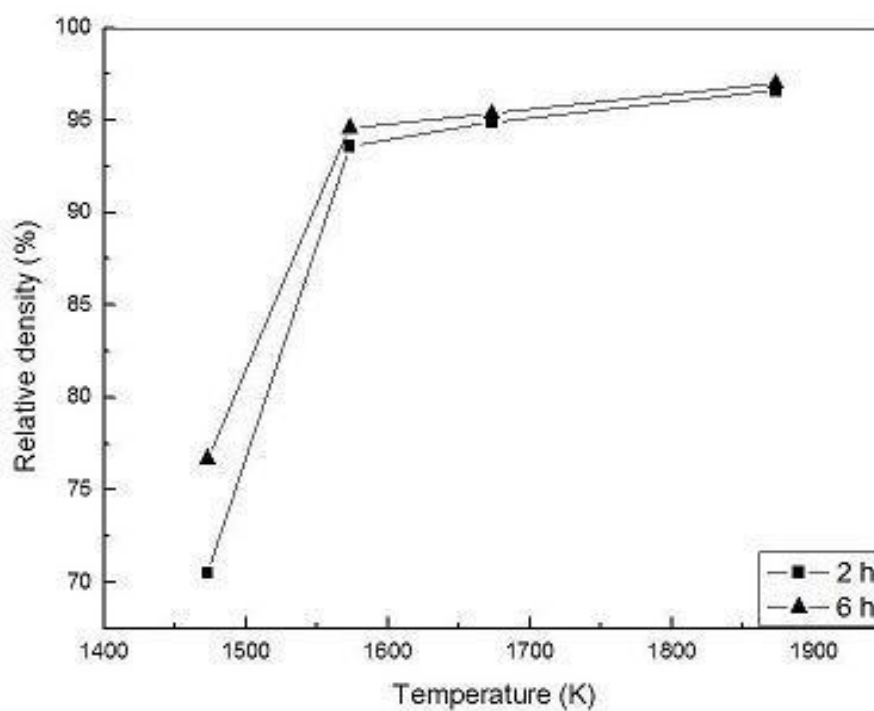

Figure 2. The relative density of 3Y-PSZ pellet sintered at various temperatures for 2 and 6 h

The variation of the Vickers hardness of the 3Y-PSZ pellet samples sintering at various temperatures for different times are listed in Table. 1. It is seen that the Vickers hardness increases with sintering temperatures and times. Moreover, when sintered at 1473 K and 1573 K for 2 h, respectively, the increment of Vickers hardness was larger than at 1473 K and 1573 K for 6 h. This result shows that the hardness is dependent on the variation of density.

Table 1. Vickers hardness for 3Y-PSZ pellet samples sintered at various temperatures for different times

Temperature (K)	Vickers hardness (HV)	
	sintered for 2 h	sintered for 6 h
1473	648	884
1573	870	999
1673	1078	1093
1773	1118	1156

Figure 3 shows the TEM microstructures and nano beam diffraction patterns (NBDP) of 3Y-PSZ pellet with sintered at 1773 K for 2 h. Fig. 3(a) and (b) shows the images of bright field (BF) and dark field (DF), it is seen that the average grain size of about 200 nm. Fig. 3(c) shows the NBDP which index corresponding to t-ZrO_2. Fig. 3(d) and (e) also shows the DF image and NBDP, respectively. The NBDP pattern which index correspond to m-ZrO_2.
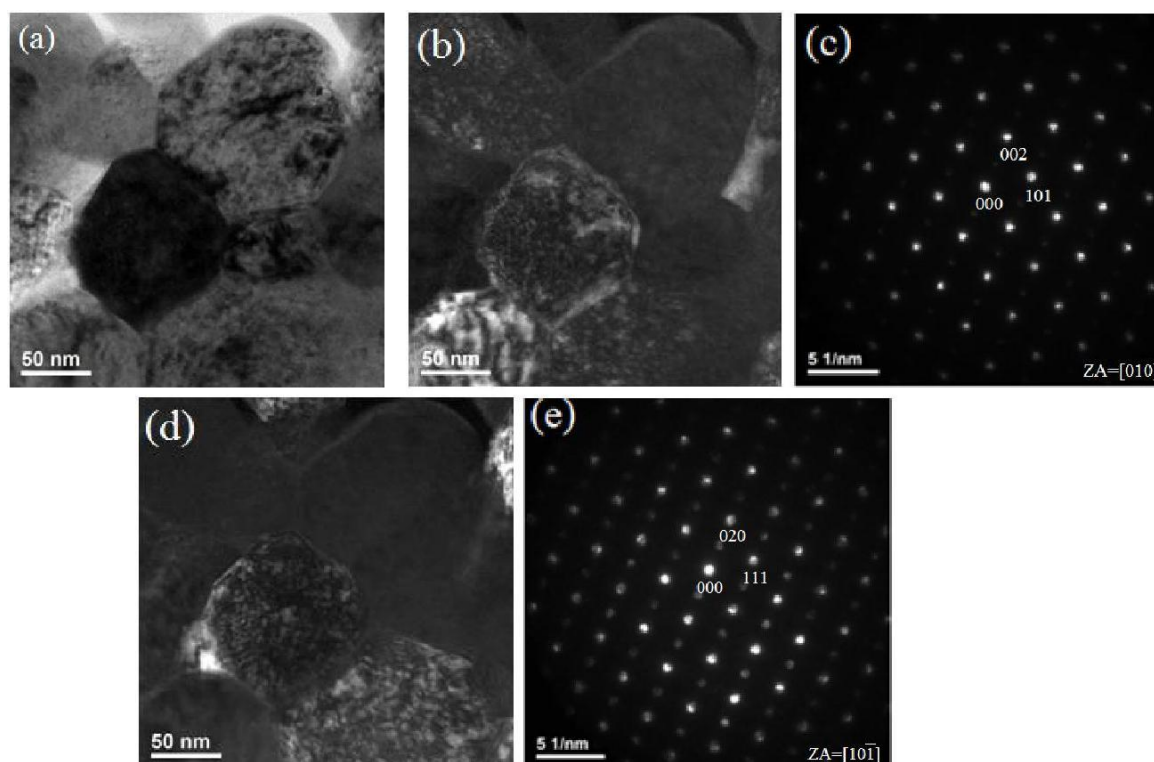

Figure 3. TEM microstructures and NBDP patterns of 3Y-PSZ pellet with 1 wt% NAS glass additive are sintered at 1773 K for 2 h: (a) BF image, (b) DF image, (c) NBDP pattern index correspond to t-ZrO_2, (d) DF image, (e) NBDP pattern index correspond to m-ZrO_2

4. Conclusion

Effect of sintering process parameters on the properties of 3Y-PSZ ceramic has been investigated using XRD, Vickers hardness test, TEM and SAED. The results are given as follows:

(1) The crystal structures of all the 3Y-PSZ sintered pellets were the phases of t-ZrO_2 and m-ZrO_2 coexist.

(2) The relative density of sintered 3Y-PSZ pellets increases from 76.7 to 97.0 % when sintering at 1473 to 1773 K for 6 h.

(3) The Vickers hardness of sintered 3Y-PSZ pellets increases with rose sintering temperatures. On the other hand, when sintering at 1473 K and 1573 K for 2 and 6 h, respectively, the increment of Vickers hardness was dependent on the variation of density.

References

[1] R.H.J. Hannink, P.M. Kelly and B.C. Muddle: J. Am. Ceram. Vol. 83 (2000) p. 461

[2] M.B. Blatz, A. Sadan and M. Kern: J. Prosthet. Dent. Vol. 89 (2003) p. 268

[3] F.F. Lange: J. Mater. Sci. Vol. 17 (1982) p. 247

[4] S. Deville, J. Chevalier, G. Fantozzi, J.F. Bartolome, J. Requena,J.S. Moya, R. Torrecillas and L.A. Diaz: J. Euro. Ceram. Vol. 23 (2009) p. 2975

[5] C. Piconi and G. Maccauro: Biomaterials Vol. 20 (1999) p. 1

[6] Y. Zhang and B.R. Lawn: J. Biomed. Mater. Res. B: Appl. Biomater. Vol. 72B (2005) p. 388

[7] S.Y. Liu and I.W. Chen: J. Am. Ceram. Soc. Vol. 74 (1991) p. 1197

[8] H. Huang: Mater. Sci. Eng. A: Struct Vol. 345 (2003) p. 155

[9] W.F. Ho, H.C. Hsu, Y.F. Peng and S.C. Wu: Ceram. Int. Vol. 37 (2011) p. 1169