Improvement on Flexural Strength of Zirconia via Three-Step Sintering Process

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Statement of Purpose: Yttria stabilized zirconia (Y-TZP) has been widely used as a biological structure material because of excellent chemical stability under physiological conditions as well as good biocompatibility. In particular, mechanical enhancement of zirconia via controlling the grain size has been studied according to the recent finding that ceramics with ultrafine grains exhibit superplastic deformation and higher hardness with increased toughness [1]. Recently, two-step sintering for Y-TZP has been proposed for the method to obtain fully-dense zirconia with ultrafine grains, where the second step only allows limited diffusion for densification, minimizing grain growth [1, 2]. However, none of those studies have reported mechanical improvement associated with reduced grain size. Thus, in this study, we have assessed mechanical properties of zirconia from various sintering processes and have introduced a three-step sintering method for further mechanical enhancement.

Methods: ZrO_2-3 mol% Y_2O_3 powder was first compacted under the uniaxial pressure of 200 MPa and subsequently densified under hydrostatic pressure of 200 MPa using Cold Isotatic Pressure (CIP) equipment. The dense green body was sintered under three conditions, at the heating rate of 5 °C/min and the cooling rate of 20 °C/min for all specimens: (1) Single-step sintering at 1450 °C for 2 h. (2) Two-step sintering with the first peak temperature of 1450 °C for 15 min followed by 30 h holding at 1150 °C. (3) Three-step sintering with additional sintering step at 1450 °C for 1 h after the two-step sintering of (2). The microstructures of sintered zirconia specimens were observed by SEM after polishing and thermal etching, and the average grain size of each sample was determined by the linear intercept method using SEM images. Relative density of each sample was calculated as compared to theoretical density of zirconia after measurement of density via Archimedes' method. The flexural strength of the specimens was measured by the biaxial bending test (ISO-6872) with a loading speed of 1 mm/min.

Results: Fig. 1 shows microstructures of the specimens with RD > 99 %, where each microstructure of three sintering conditions was found to have the average grain size of 437, 359 and 371, respectively as shown in Fig. 2. By comparing two-step and three-step sintering, the additional step of three-step sintering was found to induce further grain growth at some degree with minimal influence on densification, implying that the third step doesn't contribute to the densification process. Obviously, single-step sintering results in the largest grains, whereas two-step sintering does the finest with relatively uniform size. In Fig. 3, flexural strength of single-step sintered Y-TZP is the smallest because of larger grain size. Interestingly, as compared to the two-step sintered Y-TZP, the three-step sintered Y-TZP exhibits significant improvement of flexural strength in spite of the comparable grain size.

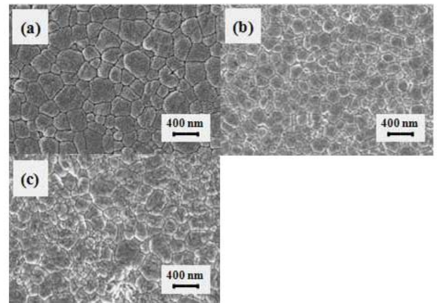

Figure 1. SEM micrographs of Y-TZP from (a) Single-step, (b) Two-step, (c) Three-step sintering

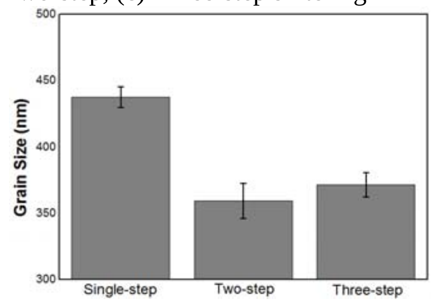

Figure 2. The average grain size of fully-dense zirconia (RD > 99%) after indicated sintering cycles

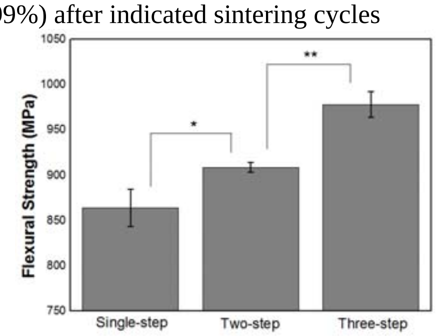

Figure 3. Flexural strength of fully-dense zirconia (RD > 99%; n=3) after indicated sintering cycles (*$p<0.05$, **$p<0.01$)

Conclusions: Two-step sintering significantly improved mechanical properties as compared to conventional single step sintering because of ultrafine grains. However, the additional three-step sintering shows further improvement of mechanical behavior with comparable grain size to one from two-step sintering. Therefore, the three-step sintering has great potential as a promising method to optimize mechanical behavior of Y-TZP for various biomedical applications.

References:

[1] M. Mazaheri et al., J. Eur. Ceram. Soc. 2008:28:2933-2939.

[2] Chen, I. W et al., Nature. 2000:404:168-171.