

## Fabrication of Continuously Porous t-ZrO<sub>2</sub> Bodies Using Carbon Powder and Evaluate Their Biocompatibility

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### 1. Introduction

Recently, yttria stabilized zirconia (t-ZrO<sub>2</sub>) has been considered for a typical bioceramic for bioimplant such as total hip prosthesis and dental materials [1] due to their good biocompatibility as well as good materials properties such as high strength, excellent chemical stability and good wear resistance [2]. Especially, t-ZrO<sub>2</sub> ceramic showed excellent fracture toughness of about 4-15MPa<sup>1/2</sup> as compared with 2-12MPa<sup>1/2</sup> for human bone, due to the presence of a phase transformation toughening mechanism [3]. The most important factors for the implant application are the biocompatibility and reasonable material properties. To improve the biocompatibility of the ceramics, the pore size, the pore shape and porosity are important factors because they are closely related to the cell attachment, growth behavior and bond strength between the tissue and the artificial implant in the human body. Suitable pore size was reported to be approximately 100-150, 140-160 and 200-1000μm in diameter [4].

In this work, the continuously porous t-ZrO<sub>2</sub> bodies were fabricated by the extrusion process using commercial t-ZrO<sub>2</sub> powder and evaluate their biocompatibility using human osteoblast like MG-63 cells and osteoclast like Raw 264.7 cells.

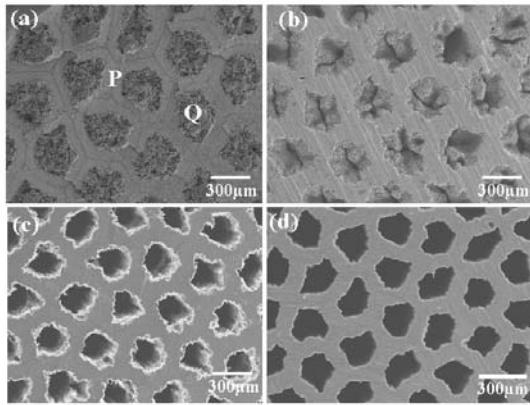
### 2. Experiment Procedure

To fabricate the continuously porous t-ZrO<sub>2</sub> ceramics, the starting t-ZrO<sub>2</sub> (about 70nm in average diameter, Tosoh Corporation, Nanyo manufacturing complex, Japan) powder, pore forming agent (carbon, about 20μm in average diameter, Aldrich Chemical Company, USA) binder (ethylene vinyl acetate, Elvax 250, Dupont, USA) and lubricant (stearic acid (CH<sub>3</sub>(CH<sub>2</sub>)<sub>16</sub>COOH, Daejung Chemicals & Metals Co., Korea) were used. Using t-ZrO<sub>2</sub>/EVA/ stearic acid with volume fraction 50/40/10 and C/EVA/ stearic acid volume fraction 50/40/10 were homogeneously mixed by shear mixer. These mixtures were

used to make the tube and rod, respectively. The tube and rod were assembled together to prepare a feed roll and extruded at 120°C to make the 2<sup>nd</sup> passed extruded filaments. To remove the polymer binder, the 1<sup>st</sup> burn-out was performed in a tube furnace with a slow heating rate (45°C/hr) up to 700°C in flowing nitrogen gas and pore forming agent was removed by 2<sup>nd</sup> burn-out process at 1000°C in air atmosphere. Finally, the sintering process was carried out from 1200°C-1600 °C for 1 hr in flowing air. Microstructure and crystal structure were examined using scanning electron microscope (SEM, JSM-635F, Jeol, Japan) and X-ray diffraction (XRD, D/MAX-250, Rigaku, Japan). Densities of porous samples were measured by the Archimedes method. The average bending strength was measured by a four-point bending test method using a universal testing machine (Unitech<sup>TM</sup>, R&B, Korea) and evaluates their biocompatibility using MG-63 human osteoblast-like cell and osteoclast cells.

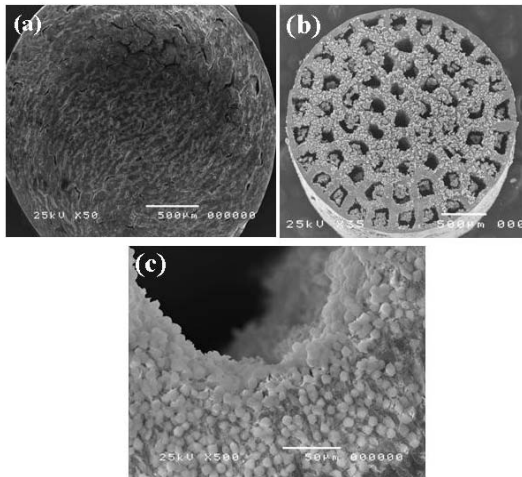
### 3. Results and Discussion

Fig. 1 shows the low magnification SEM micrographs of (a) extruded body, (b) after 1<sup>st</sup> burn-out, (c) after 2<sup>nd</sup> burn-out and (d) after sintering of continuously porous t-ZrO<sub>2</sub> bodies. In SEM image (a), the bright and dark contrasts were t-ZrO<sub>2</sub> and pore forming agent carbon, respectively. After 1<sup>st</sup> burn-out (b), the polymer binder was removed while the pore forming agent carbon was still remained. But, in the pore forming agent (carbon) region some cracks were clearly observed. During the 2<sup>nd</sup> burn-out process (c), the combustible pore-forming agent carbon was removed due to the reaction of oxygen by the formation of CO and CO<sub>2</sub>. However, after sintering, the continuously porous t-ZrO<sub>2</sub> bodies were fabricated and well controlled the pore size and shape. The pore size of 2<sup>nd</sup> passed filaments was about 260μm in diameter.



**Fig. 1. SEM micrographs of (a) extruded body, (b) after 1<sup>st</sup> burn-out, (c) after 2<sup>nd</sup> burn-out and (d) after sintering of continuously porous t-ZrO<sub>2</sub> bodies.**

Fig. 2 shows SEM micrographs of cell growing behavior of (a) human MG-63 osteoblast and (b,c) osteoclast Raw 264.7 cells on the top surface of the continuously porous t-ZrO<sub>2</sub> bodies after 7 days. After osteoblast cell loading on the top surface of the continuously porous t-ZrO<sub>2</sub> bodies, most of the cell went down through the continuous pores and a few cells were adhered on the pore frame surface. After 7 days, the MG-63 osteoblast cells were well grown in porous t-ZrO<sub>2</sub> bodies and fully covered the continuously pores as well as the frame regions of pores. However, the pebble stone like osteoclast cells were well grown and fully covered the pore frame regions (c).



**Fig. 2. SEM micrographs of (a) human MG-63 osteoblast and (b,c) osteoclast Raw 264.7 cells grown behavior on the continuously porous t-ZrO<sub>2</sub> bodies.**

#### 4. Conclusions

Continuously porous t-ZrO<sub>2</sub> bodies were fabricated by the extrusion process using combustible carbon powder as a pore-forming agent. The values of the relative density and the bending strength of continuously porous t-ZrO<sub>2</sub> bodies sintered at 1500°C were about 67% and 177MPa, respectively. From the in-vitro investigation using human osteoblast like MG-63 cells and osteoclast like Raw 264.7 cells, we confirmed that all kinds of cells were well grown and fully covered pore frame region as well as the pores.

#### 5. Acknowledgement

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#### 6. References

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