Highly Aligned Porous Alumina Ceramics by Extruding Unidirectionally Frozen Alumina/Camphene Body

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Introduction: Porous bioceramics with aligned pores have recently attracted increasing interest, since their mechanical properties can be improved significantly without sacrificing good biocompatibility [1]. One of the most promising approaches for this goal is unidirectional freeze casting of an aqueous ceramic slurry, where aligned pores can be readily produced as the replica of the ice dendrites preferentially grown during freezing [2]. More recently, camphene as an alternative freezing vehicle was also used successfully to produce highly aligned porous materials [3]. However, it is practically difficult to maintain the continuous preferential growth of dendrites during the entire process, which would limit the degree of aligned pores[3,4]. Therefore, we herein proposes a novel, simple way of producing porous alumina bioceramics with highly aligned pores by extruding a unidirectionally frozen alumina/camphene body. The pore structure of the sample produced was closely examined by scanning electron microscopy (SEM) and compressive strength tests were carried out.

Method: A ceramic slurry was prepared by mixing commercial alumina powder with molten camphene containing 3 wt% of an oligomeric polyester dispersant using ball-milling at 60°C for 24 h. In particular, polystyrene polymer with a content of 10 vol% in relation to the alumina powder was added to improve the green strength of the frozen sample. The prepared slurry was cast into a mold attached to a copper plate as a cooling part and then placed at ~3°C for 1h to allow the unidirectional freezing of the slurry. After which, the frozen sample was extruded at a constant speed of 5 mm/min, followed by freeze drying to completely remove the frozen camphene dendrites. The green samples were heat-treated at 1450°C for 3 h to sinter the alumina walls. The pore structure of the sample was observed by scanning electron microscopy (SEM). The porosity of the samples was calculated from its dimensions and weight. The pore size and wall thickness were also analyzed from the SEM images. In addition, compressive strength tests were carried out.

Results and Discussion: A highly aligned porous alumina ceramic was produced successfully by extruding a frozen alumina/camphene slurry. Before extrusion, the sample showed aligned pores which were formed as the replica of the camphene dendrites which grew in a preferential orientation that was parallel to the direction of unidirectional freezing (Fig. 1 (A)). These pores could be extensively elongated during extrusion (Fig. 1 (B)), which would, consequently, lead to the creation of highly aligned pores (Fig. 1 (C)), suggesting the formation of the aligned pores. In addition, the honeycomb-like pores were formed normal to the direction of freezing (Fig. 1 (D)). However, the pore size decreased remarkably from ~ 130 to 13 μm after extrusion, while showing the similar porosities, as summarized in Table I. The achievement of the aligned pores endowed the porous alumina ceramic with a high compressive strength of 0.28 ± 0.08 MPa, when compressed parallel to the direction of pore alignment (Table II). It should be noted that these aligned porous ceramics can be used to produce hybrid composites which can resemble the architecture of nacre with unusually high strength and toughness.

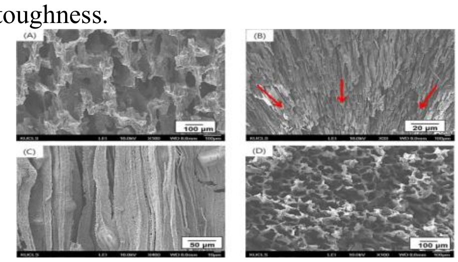

Figure 1. SEM images of the highly aligned porous bioceramics during process: before extrusion (A), during extrusion (B), after extrusion ((C),(D)).

Table I. Total porosity, pore size and wall thickness of the porous alumina ceramic.

Extrusion	Porosity[vol%]	Pore size[μm]	Wall size[μm]
Before	83	130	18.2
After	82	13.2	2.16

Table II. Compressive strengths of the samples compressed parallel and normal to the direction of pore alignment.

Direction	Strength [MPa]
Parallel	0.28 ± 0.08
Normal	0.05 ± 0.01

Conclusions: We fabricated highly aligned porous alumina ceramic by extruding a unidirectionally frozen alumina/camphene body, where preferentially grown camphene dendrites could be extensively elongated during extrusion. The fabricated sample showed a highly aligned porous structure with a pore size of ~ 13 μm and a porosity of 82 vol%. The compressive strength of the sample was as high as 0.28 ± 0.08 MPa, when compressed parallel to the direction of pore alignment, owing to the construction of the highly aligned pores

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