



## Reverse freeze casting: A new method for fabricating highly porous titanium scaffolds with aligned large pores

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### ARTICLE INFO

#### Article history:

Received 19 October 2011

Received in revised form 21 January 2012

Accepted 5 March 2012

Available online 13 March 2012

#### Keywords:

Scaffold

Freeze casting

Aligned pores

Titanium (Ti)

Mechanical properties

### ABSTRACT

Highly porous titanium with aligned large pores up to 500  $\mu\text{m}$  in size, which is suitable for scaffold applications, was successfully fabricated using the reverse freeze casting method. In this process we have newly developed, the Ti powders migrated spontaneously along the pre-aligned camphene boundaries at a temperature of 45.5  $^{\circ}\text{C}$  and formed a titanium–camphene mixture with an aligned structure; this was followed by freeze drying and sintering. As the casting time increased from 24 to 48 h, the initial columnar structures turned into lamellar structures, with the porosity decreasing from 69 to 51%. This reduction in porosity caused the compressive yield strength to increase from 121 to 302 MPa, with an elastic modulus of the samples being in the range of 2–5 GPa. In addition, it was demonstrated that reverse freeze casting can also be successfully applied to various other raw powders, suggesting that the method developed in this work opens up new avenues for the production of a range of porous metallic and ceramic scaffolds with highly aligned pores.

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### 1. Introduction

Dense ceramic and metallic orthopedic biomaterials generally have an elastic modulus which is five to ten times higher than that of the human cortical bone. Owing to such a mechanical mismatch, the stiffer implant material carries the greater part of the load, which usually leads to bone resorption and the loosening of the implant [1]. This undesirable phenomenon, called the “stress shielding effect”, needs to be eliminated to improve the long-term fixation and avoid revision surgery. As a primary solution to this problem, a number of surface treatment techniques, such as sand blasting [2], plasma spray coating [3,4], and micro-arc oxidation [5,6], have been considered for producing a rough surface which can enhance the physical fixation of the implant. However, after evaluating these methods it was found that they do not solve the fundamental problem [1].

In recent years, various porous materials have attracted much attention in the context of tissue engineering with bone or cartilage [7,8]. Porous materials are lighter than the existing dense implants and generally have a lower elastic modulus, which can reduce the stress shielding effect [1,9]. The structure of these materials must be highly porous – with porosity in excess of 50% and

interconnected open pores in the size range of 100–500  $\mu\text{m}$  to allow bone growth into the scaffold [8,10–13]. In particular, it was reported that average pore sizes larger than 150  $\mu\text{m}$  provide better conditions for bone formation and vascularization [14]. However, highly porous materials (over 50% porosity) generally exhibit poorer mechanical properties than human cortical bone, which can cause fracture of the implant during the healing stage, even with metallic materials [1,15–29]. This is particularly true in the case of compressive loading. Therefore, the development of superior materials showing enhanced strength at the same porosity level is required.

Since the mechanical properties of porous materials depend mainly on their pore structure (size and shape of the pores, level of porosity and degree of pore alignment), a variety of methods of manufacturing strong porous materials have been reported. These include the rapid prototyping method [30,31], unidirectional freeze casting [32–45], the replication method [17,46–48], the space-holder method [49], the ionotropic gelation of alginate [50], and further techniques.

Unfortunately, the evaluation of most porous ceramic materials shows that commonly they cannot be employed in load-bearing parts because of their brittleness. Besides, porous ceramics typically exhibit a lower compressive strength and poorer overall compressive behavior than metallic materials, as crack initiation sites at the intersections of the struts are more detrimental in ceramics.

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