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# Mechanical properties of porous ceramic scaffolds: Influence of internal dimensions

I. Sabree, J.E. Gough, B. Derby\*

School of Materials, University of Manchester, Oxford Road, Manchester M13 7PL, UK Received 20 February 2015; received in revised form 6 March 2015; accepted 9 March 2015

#### **Abstract**

Highly porous ceramic scaffolds have been fabricated from a 70% SiO<sub>2</sub>–30% CaO glass powder using stereolithography and the lost-mould process combined with gel-casting. After sintering at 1200 °C the glass crystallised to a structure of wollastonite and pseudowollastonite grains in a glassy matrix with a bulk porosity of 1.3%. All scaffolds had a simple cubic strut structure with an internal porosity of approximately 42% and internal pore dimensions in the range 300–600  $\mu$ m. The mean crushing strength of the scaffolds is in the range 10–25 MPa with the largest pore sizes showing the weakest strengths. The variability of scaffold strengths has been characterised using Weibull statistics and each set of scaffolds showed a Weibull modulus of  $m \approx 3$  independent of pore size. The equivalent strength of the struts within the porous ceramics was estimated to be in the range 40–80 MPa using the models of the Gibson and Ashby. These strengths were found to scale with specimen size consistent with the Weibull modulus obtained from compression tests. Using a Weibull analysis, these strengths are shown to be in accordance with the strength of 3-point bend specimens of the bulk glass material fabricated using identical methods. The strength and Weibull modulus of these scaffolds are comparable to those reported for other porous ceramic scaffold materials of similar porosity made by different fabrication routes. © 2015 Published by Elsevier Ltd and Techna Group S.r.l.

Keywords: Porous ceramics; Stereolithography; Gel casting; Weibull statistics

#### 1. Introduction

The scaffold is a key concept in tissue engineering. This is a porous structure that acts as a substrate, upon the surface of which cells adhere and grow. The scaffold provides structural and mechanical support as well as the surface for cell growth. Its presence allows cells to generate the biological structural components of the extracellular matrix (ECM) in culture conditions. After a suitable period of culture and after implantation into a host, sufficient ECM will produce an appropriate tissue to provide mechanical integrity and the scaffold will either be harmlessly incorporated or degrade, dissolve and ultimately be excreted. For many applications, especially in the area of bone tissue engineering, this scaffold will be fabricated from an inorganic ceramic or glass [1]. The material composition of the scaffold, its gross architecture (dimensions of

http://dx.doi.org/10.1016/j.ceramint.2015.03.044 0272-8842/© 2015 Published by Elsevier Ltd and Techna Group S.r.l. its walls, pores and channels), the material microstructure and crystallinity all play a part in controlling the local environment and well being of the cells located within it [2–4].

The appropriate architecture and dimensions of a suitable scaffold are determined by a number of requirements for biocompatibility. An important role of the scaffold is to provide a temporary home for the growth and culture of cells in a bioreactor environment. Within a living tissue, cells are maintained in good health by a supply of oxygen and nutrients and the removal of carbon dioxide and waste through the capillary network. Each cell must be sufficiently close to the network to allow diffusional transport within an appropriate time scale. By analogy, all cells within the scaffold must have a similar access requirement, and this is normally achieved by providing a structure with a high level of porosity of an appropriate dimension to allow the uninterrupted flow of fluid within a bioreactor. Hutmacher has reviewed the architectural and topological requirements of scaffold design for tissue engineering cell culture [2].

<sup>\*</sup>Corresponding author.

Highly porous scaffold designs possess a large surface area, which favours cell attachment and growth. Porosity, ranging from 40% to 90% in a variety of materials, encourages osteointegration with the implant surface and promotes adhesion of the implant [5]. In addition, a porous surface enhances a mechanical interlock between the scaffold and host tissue [6]. The mean pore size of a scaffold controls cell adhesion, migration, tissue formation, nutrient and oxygen access as well as waste removal [7]. Hulbert defined a minimum pore size for a scaffold at 100 µm [8]. However, later studies have shown that better osteogenesis occurs with a pore size  $> 300 \,\mu m$ [5,6]. In addition, the pores should be interconnected. The interconnection size will be smaller than the pore size but if must be sufficient to permit cell migration, communication between cells and ECM formation between the pores. It is well known that the porosity of a foamed structure has lower mechanical properties (elastic constants, ultimate strength, fracture resistance) than the equivalent bulk material and that these reduced properties are a function of the relative density (1 – porosity) of the foam [9]. Thus, when using porous structures for tissue engineering scaffolds, the structure must retain sufficient mechanical properties to fulfil the requirements of structural integrity once implanted in host tissue. For bone tissue engineering applications the consensus is that ceramic and inorganic materials should have mechanical properties similar to that of bone. However, the mechanical properties of bone have a large range of reported values and depend on the local density of the tissue and testing environment. For example, cortical bone has a reported Young's modulus in the range 1-20 GPa and a strength range of 1-100 MPa [10], with the equivalent values for cancellous (trabecular) bone of Young's modulus 0.1-1.0 GPa and strength 1–10 MPa [11]. Despite the large range in the reported mechanical properties of bone, these act as a guide to the required mechanical properties of a scaffold.

There have been a number of different microstructures or architectures of highly porous ceramic and glass scaffolds that have been used for tissue engineering applications. These can be broadly divided into two classes: random porosity and designed porosity. Random pore architectures display a structure showing no significant long range order or alignment to the pore distribution, with pore size and shape showing significant variation around the mean values. Such structures are generally termed ceramic foams and they can be achieved by methods such as casting a slurry around a sacrificial polymer foam template, using poro-generators such as a soluble salt or polymer microbeads and by the addition of surfactant foaming agents and stabilisers prior to gaseous foaming [1,12–15]. Although it is possible to control some aspects of the foam structure (mean cell size and wall thickness), they are random on a local scale and any microstructural control is of the average properties of the structure. A second set of manufacturing processes must be used in order to achieve a more precise control of porous ceramic scaffolds and define both pore and interconnection size. These are variously described as rapid prototyping, additive manufacture or 3D printing. These manufacturing technologies fabricate

structures with a spatial resolution or feature size  $> 30 \mu m$  and can be used to explicitly define and fabricate a bespoke structure [16,17].

The basic requirements of a scaffold material are high cell/ tissue biocompatibility, non-toxicity, capability of promoting cell proliferation and differentiation, and sufficient mechanical properties. Bioactive glasses have remarkable advantages such as good biocompatibility, osteoproductivity and osteoconductivity [18]. Various studies have determined that ionic dissolution products from bioactive glasses can enhance osteogenesis by activating genes found in osteoblasts and stimulating regeneration of bone tissue [19–21]. In this study we have selected a phosphate-free bioactive glass of composition 70% SiO<sub>2</sub>-30% CaO. This composition is known to support osteoblast growth and induce differentiation when used to form scaffold materials [22]. We have chosen the process of indirect manufacture to form scaffold structures based on the 70% SiO<sub>2</sub>-30% CaO glass composition. This uses the additive manufacture route of stereolithography to fabricate moulds that define the scaffold internal architecture and feature dimensions. These moulds are used to manufacture the scaffolds using the gel-casting process following the general procedures originally pioneered by Halloran and co-workers [23]. A range of scaffolds have been manufactured with different pore sizes but the same overall porosity to explore how the pore size influences scaffold strength.

#### 2. Methods

#### 2.1. Scaffold materials and fabrication

Phosphate-free bioactive glass powder, of composition 70%  $SiO_2$ –30%  $CaO_2$ , was provided by Julian Jones and Gowishan Poologasundarampillai (Imperial College, London, UK). This had an as-received mean particle size of approximately 50  $\mu$ m, which was reduced to a size suitable for processing and sintering by milling. Glass powder (150 g) was mixed with 250 ml distilled water and two to three drops of Dolapix CE64 added (Zschimmer & Schwarz, Lahnstein, Germany) before processing in an attrition mill using ZrO<sub>2</sub> milling media (Szegvari Attritor System, Union Process, Akron, OH, USA). At the end of the milling process the slurry was separated from the milling media and freeze dried (Micro Modulyo, Edwards, Hastings, UK). The freeze dried powder had a mean particle size of 3.2  $\mu$ m measured by light scattering (Mastersizer Plus, Malvern Instruments, Great Malvern, UK).

To manufacture the scaffolds we used a modification of the "lost-mould" process for ceramic manufacture. Here a polymer mould is used to define the complex shape of a ceramic (or glass) body prior to high temperature sintering. The mould is filled with fine particles of the ceramic or glass in suspension in a fluid of cross-linkable oligomers. The fluid is gelled by an external stimulus, usually mild heating to form a composite block of the mould and the ceramic in a polymer matrix (gel casting). The polymers are removed by a heat treatment in air as part of the thermal cycle during sintering. Gel casting and

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