

Cellular Ti–6Al–4V structures with interconnected macro porosity for bone implants fabricated by selective electron beam melting

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Received 29 October 2007; received in revised form 15 February 2008; accepted 20 March 2008

Available online 10 April 2008

Abstract

Selective electron beam melting (SEBM) was successfully used to fabricate novel cellular Ti–6Al–4V structures for orthopaedic applications. Micro computer tomography (μ CT) analysis demonstrated the capability to fabricate three-dimensional structures with an interconnected porosity and pore sizes suitable for tissue ingrowth and vascularization. Mechanical properties, such as compressive strength and elastic modulus, of the tested structures were similar to those of human bone. Thus, stress-shielding effects after implantation might be avoided due to a reduced stiffness mismatch between implant and bone. A chemical surface modification using HCl and NaOH induced apatite formation during in vitro bioactivity tests in simulated body fluid under dynamic conditions. The modified bioactive surface is expected to enhance the fixation of the implant in the surrounding bone as well as to improve its long-term stability.

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Keywords: Titanium alloys; Surface modification; Bioactivity; Selective electron beam melting; Porous structures

1. Introduction

Pure titanium and some of its alloys have been extensively used as “load-bearing” implants for biomedical applications, due to their high strength-to-weight ratio, corrosion resistance in the physiological environment, fatigue resistance and low elastic modulus [1–3].

Several studies have demonstrated that a biomechanical mismatch between an implant and the surrounding tissue may lead to stress-shielding phenomena which induce an unfavourable stress distribution at the bone–implant interface, retarding both bone healing and remodelling [4,5]. Thus, a major goal within the biomedical community is to develop new metallic biomaterials for load-bearing orthopaedic and dental applications with an elastic modulus similar to that of human bone.

Bone consists of the outer cortical bone, a dense structure with high mechanical strength, and the inner trabecular bone, a network of struts enclosing large voids with 55–70% interconnected porosity. Porous metals with an interconnected pore structure are of particular interest for orthopaedic implant applications due to their potential ability to facilitate tissue ingrowth [6]. An interconnected pore system with pore diameters in excess of 100 μ m is required for cell penetration, tissue ingrowth, vascularization and nutrient delivery to the centre of the regenerating tissue [7,8]. In addition, porous metals represent a promising means of reducing stiffness mismatch and avoiding stress-shielding effects [6]. Open-cell titanium structures can be fabricated by a number of different techniques, e.g. controlled sintering of powder preforms [5], solid-state foaming by superplastic expansion of argon-filled pores [9] or polymeric foam replication [10]. However, these manufacturing techniques have limitations concerning the control of the outer shape and the pore structure. In addition, porous titanium foams have been prepared from titanium powders

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with help of space holders [11]. According to micro computer tomography (μ CT), this processing technique resulted in porous structures with a large spread of pore sizes in the range 200–500 μ m [12]. Recently, Li et al. published a rapid prototyping technique to create porous Ti–6Al–4V implants with controlled size, pore shape and distribution using a three-dimensional (3D) printing of slurries [13,14]. In this case the preparation of the slurry and the shrinkage of the porous structures after sintering seem to be critical steps.

Selective electron beam melting (SEBM) is a new additive manufacturing technique with high capability for the fabrication of porous metals with well-defined cellular structures [15]. The basic principle of this technology is the generation of structures by the selective melting of discrete powder layers by an electron beam under vacuum. With this approach a huge variety of cellular metal structures with different densities and cell morphologies is feasible. Due to its high affinity for atmospheric gases such as oxygen and nitrogen, fabrication under vacuum is of particular importance for titanium. The absorption of atmospheric gases should be effectively prevented because this might lead to a reduction in the ductility of titanium.

It is well established that the osseointegration process is affected by surface modifications in terms of chemical and physical properties [16]. Two directions have been reported for titanium and its alloys in order to improve their bone-bonding ability. Among coating techniques, plasma spraying of hydroxyapatite is the most frequently used in clinical practice. However, coating methods still encounter some general problems, including the lack of adherence to the substrate and non-uniformity of the layer thickness. Another possibility to modify titanium surfaces is represented by chemical treatments in order to obtain OH-groups that have been described as favourable to enhanced osseointegration [17,18].

The aim of this study was to fabricate bioactive cellular Ti–6Al–4V structures with a controllable interconnected porosity by SEBM. The mechanical properties of these structures were examined under compression load and compared with the properties of human bone. Surface modifications were performed by a wet chemical treatment in HCl and NaOH, and the bioactivity of the thus prepared structures was evaluated by testing the *in vitro* apatite formation in dynamic simulated body fluid (SBF).

2. Materials and methods

2.1. Fabrication of cellular titanium structures

Samples of two different cellular titanium structures with a nominal layer thickness of 100 μ m were fabricated in-house by SEBM on a machine supplied by a commercial vendor (EBM S12, Arcam AB, Sweden).

In Fig. 1 the general procedure of the additive manufacturing by SEBM is depicted. Firstly, a 3D computer-aided design (CAD) model is sliced into layers with constant thicknesses to provide layer information. The SEBM pro-

cess starts with the homogeneous application of a layer of metal powder on a process platform. After a preheating step, an electron beam scans the powder layer and creates a cross-section of the part by fusing the loosely joined powder particles. Subsequently, the process platform is lowered by the thickness of one layer, a new powder layer is applied and the process is repeated until the whole part has been built. The process is performed under vacuum (10^{-4} to 10^{-5} mbar). Since the generation process takes place in a powder bed, the metal powder that is not molten during the process acts as a support material for the parts.

The two structures used in this study vary with respect to manufacturing method, porosity and cell structure. The first structure, named the diamond structure, is based on the CAD model of diamond lattice, where each atom is surrounded tetrahedrally by four other atoms. The second structure, named the hatched structure, was generated by scanning the powder layers with the electron beam in parallel lines with constant spacing (here 1.0 mm). The scanning direction was altered by 90° every eighth layer. Due to the small spot size of the beam, the molten lines do not overlap and a porous 3D structure is produced. In this case the cell structure is not determined by the CAD model, which only defines the outer specimen dimensions, but by the process parameters. Both manufacturing methods for cellular titanium and the SEBM process in general are explained in detail by Heintl et al. [15].

Spherical titanium powder of the alloy Ti–6Al–4V with a mean particle size of 70 μ m was used for the generation of the samples. The chemical composition of the titanium powder used and of the generated structures corresponds to the specification ASTM F 1108 for Ti–6Al–4V alloy castings for surgical implants. Two different sample geometries were fabricated to achieve the requirements for the conducted compression and bioactivity tests. The samples for the compression tests were generated oversized, cleaned by blasting with titanium powder and machined to cuboids with coplanar faces and dimensions of $15 \times 15 \times 23$ mm³. The samples for the chemical surface modification and the subsequent bioactivity tests were cubic with an edge length of 10 mm. They were also cleaned by powder blasting but not post-worked.

2.2. Chemical surface modification

Prior to chemical pretreatments the generated cellular titanium samples were sonicated with isopropanol, then rinsed with double-distilled water and dried in air. The surface modification was performed by etching of the samples in 37% HCl under an argon atmosphere for 90 min at 50 °C and subsequently for 60 min at 40 °C. Afterwards all specimens were soaked in 10 M NaOH aqueous solution at 60 °C for 24 h, washed with distilled water and dried at 100 °C [17].

Cellular titanium samples with and without treatment in HCl and NaOH were immersed in a mixture of 1.6 M ammonium chloride solution and 0.13 M zinc chloride

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