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Structural, mechanical and in vitro characterization of individually structured Ti–6Al–4V produced by direct laser forming

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Abstract

Direct laser forming (DLF) is a rapid prototyping technique which enables prompt modelling of metal parts with high bulk density on the base of individual three-dimensional data, including computer tomography models of anatomical structures. In our project, we tested DLF-produced material on the basis of the titanium alloy Ti–6Al–4V for its applicability as hard tissue biomaterial. To this end, we investigated mechanical and structural properties of DLF-Ti–6Al–4V. While the tensile and yield strengths of untreated DLF alloy ranged beyond 1000 MPa, a breaking elongation of $6.5 \pm 0.6\%$ was determined for this material. After an additional post-DLF annealing treatment, this parameter was increased two-fold to $13.0 \pm 0.6\%$, while tensile and yield strengths were reduced by approx. 8%. A Young's modulus of 118.000 ± 2.300 MPa was determined for post-DLF annealed Ti–6Al–4V. All data gained from tensile testing of post-DLF annealed Ti–6Al–4 V matched American Society of Testing and Materials (ASTM) specifications for the usage of this alloy as medical material. Rotating bending tests revealed that the fatigue profile of post-DLF annealed Ti–6Al–4 V was comparable to casted/hot isostatic pressed alloy. We characterized the structure of non-finished DLF-Ti–6Al–4V by scanning electron microscopy and observed a surface-associated layer of particles, which was removable by sandblasting as a finishing step. We manufactured porous specimens with nominal pore diameters of 500, 700 and 1000 μm. The diameters were reduced by the used DLF processing by approx. 300 μm. In an in vitro investigation, we cultured human osteoblasts on non-porous and porous blasted DLF-Ti–6Al–4V specimens to study morphology, vitality, proliferation and differentiation of the cells. The cells spreaded and proliferated on DLF-Ti–6Al–4V over a culture time of 14 days. On porous specimens, osteoblasts grew along the rims of the pores and formed circle-shaped structures, as visualized by live/dead staining as well as scanning electron microscopy. Overall, the DLF-Ti–6Al–4V approach proved to be efficient and could be further advanced in the field of hard tissue biomaterials.

Keywords: Titanium alloy; Rapid prototyping; Structure; Mechanical properties; Osteoblast; Cell spreading

1. Introduction

Rapid prototyping (RP), also known as solid freeform fabrication, is a strategy to directly generate physical objects with defined structure and shape on the basis of virtual 3D model data. Among diverse established RP technologies, selective laser sintering (SLS) [1] or selective laser powder remelting/direct laser forming (DLF) [2] offers the advantage to make use of an extended range of basic materials including polymers,

Abbreviations: ASTM, American Society for Testing and Materials; DLF, direct laser forming; HIP, hot isostatic pressing; HOB, human osteoblasts; ILT, Fraunhofer Institute of Laser Technology; PBS, phosphate-buffered saline; RP, Rapid prototyping; SEM, scanning electron microscopy; SLS, Selective Laser Sintering; XTT, sodium 3′-[1-(phenylaminocarbonyl)-3, 4-tetrazolium]-bis (4-methoxy-6-nitro) benzene sulfonic acid hydrate

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metals and ceramics. Comparing SLS and DLF techniques, SLS results in objects with sub-optimal bulk density due to partial melting and immediate sintering of the particles of the basic material. During DLF, on the other hand, the basic material particles are completely melted and fused in the laser focus resulting in objects with a density of almost 100%, resulting in a higher mechanical strength.

In principle, the DLF process can be subdivided into the following steps (Fig. 1). The structural information of the given 3D model is processed layer-wise. To this end, the model is split into layers with a defined thickness. In the production unit, the structure of the respective layer is selectively melted into a powder bed of the chosen material by a scanning laser beam. After the short exposure to the laser spot, the molten zones quickly solidify. In the next step, the production platform is lowered one layer thickness, a new powder layer is spread and scanned. The scanning orientation is altered by 90° after each layer. This process is repeated until the complete batch of layers has been transferred, resulting in a solid analog of the original 3D model, which usually only requires minimal surface finishing. Excessive basic material can be saved and reused, which additionally reduces manufacturing costs.

The prospect of instantaneously generating tailored parts via SLS/DLF has aroused much interest in the field of orthopedical and trauma surgery, where prostheses and implants have to be individually shaped in many cases. This is conventionally achieved by machining (e.g. milling, turning), which is time- and material-consuming and does not allow the realization of complex volumetric pore structures. With SLS/DLF technology, on the other side, parts with user-defined complexity can be fabricated almost realtime. The required 3D models can be directly derived from multiplanar 3D imaging of anatomical structures.

While SLS-fabricated implants on the basis of polymers, polymer/ceramic composites and ceramics are investigated since the 1990s [3–6], the realization of metal-based implants by DLF is a more recent approach. At the Fraunhofer Institute of Laser Technology (ILT), Aachen, a DLF strategy has been developed to manufacture hard tissue implants on the basis of titanium and its alloys [7]. In our present investigation, DLF titanium with various structures, porosities and post-DLF treatments is produced and investigated with regard to hard tissue substitution, which comprises structural characterization, mechanical testing as well as in vitro investigation. The aim of the project is to help establish the DLF technique as an alternative to conventional manufacturing in the field of metallic hard tissue biomaterials. In this publication, we present structural and mechanical properties of DLF-fabricated titanium including tensile strength, rotating bending fatigue and Young's modulus. As in vitro model, we used human osteoblast culture to investigate spreading, vitality, proliferation and differentiation of the cells on DLF titanium.

2. Materials and methods

2.1. DLF production

Ti–6Al–4V-powder with a particle size of 25–45 μm was used as basic material. Processing was carried out in an argon atmosphere using a Nd:G laser system. For mechanical testing, round tensile specimens according to DIN 10002-1 with diameters of 4 mm and lenghts of 20 mm (overall length: 54 mm) were fabricated. Additionally, for fatigue property testings, round specimens according to DIN 50113-A with diameters of 4 mm and lenghts of 60 mm were manufactured. For the fabrication of specimens for mechanical testing the DLF process was set to exclusively produce dense parts (>99.5%). For tensile testing, some of the specimens underwent an annealing heat treatment which was carried out at 950 °C for 30 min in order to homogenize the metallic microstructure. The specimens were built with an oversize of 1 mm in diameter. Adjacently finish turning was conducted in order to match standard surface and tolerance requirements. Both types of round specimen were built up in a lying position which means that their longitudinal axis was parallel to the substrate's surface. For the in vitro experiments, discs with a thickness of 2 mm and a diameter of 20.5 mm were manufactured. Porosity was modelled according to a regular offset array of parallel cylindrical pores.

2.2. Mechanical testing

For all mechanical tests, specimens were DLF-processed in oversize and turned to match the appropriate dimensions. Tensile testing was conducted on the basis of DIN 10002-1. The main focus of mechanical testing was to determine basic material properties to match with American Society for Testing and Materials (ASTM) standards required for Ti–6Al–4V implants [8]. Untreated and post-DLF annealed DLF-Ti–6Al–4V was tested and compared to the literature results for wrought annealed Ti–6Al–4V. For each condition, three specimens were tested.

In order to determine fatigue properties of DLF-Ti–6Al–4V under standard geometric and environmental conditions 28 specimens were fabricated in charges of four pieces. All 28 specimens were annealed and adjacently turned into shape. Subsequent fatigue testing was carried out according to DIN/EN 50113 rotating bending fatigue testing standards at 50 Hz testing frequency. In each charge specimens were tested until failure starting with constant stresses from 500 to 700 Mpa. Moving from one specimen to the next, stresses were reduced successively to 350–450 Mpa. Using this procedure, a complete array of stresses and corresponding cycles to failure was covered with up to 1×10^7 cycles.