



## Quasi-static torsional deformation behavior of porous Ti6Al4V alloy

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### ABSTRACT

Laser processed Ti6Al4V alloy samples with total porosities of 0%, 10% and 20% have been subjected to torsional loading to determine mechanical properties and to understand the deformation behavior. The torsional yield strength and modulus of porous Ti alloy samples was found to be in the range of 185–332 MPa and 5.7–11 GPa, respectively. With an increase in the porosity both the strength and the modulus decreased, and at 20% porosity the torsional modulus of Ti6Al4V alloy was found to be very close to that of human cortical bone. Further, the experiments revealed clear strain hardening and ductile deformation in all the samples, which suggests that the inherent brittleness associated solid-state sintered porous materials can be completely eliminated via laser processing for load bearing metal implant applications.

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### 1. Introduction

Metallic biomaterials currently in use for load-bearing orthopedic applications are bioinert and lack sufficient osseointegration for implant longevity [1]. One consideration to improve the healing process is focused on the use of porous metals, which can support tissue adhesion, growth and osseointegration. Moreover, porous metals can eliminate problems associated with stress shielding by tailoring their elastic modulus to match that of natural bone [1–5]. Many researchers have focused on obtaining desired mechanical properties in porous metallic biomaterials by controlling porosity characteristics via a variety of fabrication techniques [3,6–9]. While these studies provide good understanding of deformation and mechanical behavior of porous metals under tensile and compressive loads, the results may not be directly applicable to the deformation behavior and mechanical properties of porous metals under other modes of loading such as bending and torsion. Despite their physiological and mechanical relevance, the possible influence of highly complex *in vivo* loading conditions on the deformation behavior of porous metallic biomaterials has rarely been considered. Although some studies report quasi-static and dynamic deformation of dense Ti6Al4V alloy under torsional loading [10–12], in particular, work on torsional behavior of porous Ti6Al4V alloy with clinical relevance is rather scarce. Therefore, in the present work, we have evaluated the influence of porosity (0 to 20%) on the mechanical

properties and deformation behavior of laser processed Ti6Al4V alloy under torsional loading. This article also highlights the importance of laser processing, where the porosity forms as a result of localized melting and subsequent solidification, in contrast to solid-state sintering in the powder metallurgical route – leading to brittleness and loss of physical properties [13–15].

### 2. Materials and methods

Ti6Al4V alloy powder (Advanced Specialty Metals Inc., NH, USA) with a size range of 50–150  $\mu\text{m}$  was used to prepare porous samples using Laser Engineering Net Shaping—LENS™ 750 system (Optomec Inc., Albuquerque, NM, USA). Detailed description and capabilities of LENS™ process can be found elsewhere [3,7–9]. Our earlier work [5] showed that the modulus of laser processed Ti6Al4V alloy samples with a total porosity >25% was less than 10 GPa and are not suitable for direct load bearing implant applications though they may be used as coatings or scaffolds. Since the focus in this paper is to understand the influence of porosity on torsional deformation under load bearing environment, porous Ti6Al4V alloy samples with 0%, 10% and 20% total porosity were fabricated using (i) 350 W laser power, 17 mm  $\text{s}^{-1}$  scan speed, and 12 g  $\text{min}^{-1}$  powder feed rate, (ii) 300 W, 15 mm  $\text{s}^{-1}$ , and 20 g  $\text{min}^{-1}$ , and (iii) 250 W, 20 mm  $\text{s}^{-1}$ , and 23 g  $\text{min}^{-1}$ , respectively.

Samples for torsion tests with 12 mm square ends and  $\phi$  10 mm in the gauge length (35 mm) were prepared directly from a 3-dimensional computer aided model. As-fabricated samples were tested at room temperature for their torsional properties and deformation behavior using a 220 Nm torsion testing machine (Instron-55 MT, Norwood, MA). All samples were tested until failure or 40% drop in torque at a torsional

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speed of  $45^\circ \text{min}^{-1}$ . From the torque – degrees of rotation data recorded during the test, torsional yield strength, modulus, maximum shear stress and strain were calculated and an average of three tests (for each porosity) is reported along with standard deviation. Quasi-static compression tests for mechanical property evaluation were also carried out using a servo-hydraulic MTS (axial/torsion materials test system) machine with 250 kN capacity at a strain rate of  $10^{-3} \text{s}^{-1}$ . Young's modulus and 0.2% proof strength were determined from the stress–strain plots derived from load–displacement data recorded during compression testing. A regression analysis was performed on all test data and  $p < 0.05$  was considered statistically significant. The fractured surfaces of torsion samples were studied using field-emission scanning electron microscopy (FEI – Quanta 200F) to understand the influence of porosity on the deformation and failure mechanisms. Cross-sectional microstructures of the samples were also examined using FE-SEM. Vickers microhardness measurements were also made on the as-fabricated porous Ti6Al4V alloy samples using a 500 g load for 15 s, and the average value of 10 measurements was reported.

Finally, to ensure that laser processing does not have any toxic influence on Ti6Al4V alloy samples, all the samples were evaluated for their *in vitro* cytotoxicity using MTT assay. All samples were sterilized by autoclaving at  $121^\circ \text{C}$  for 20 min. In this study, the cells used were an immortalized, cloned osteoblastic precursor cell line 1 (OPC1), which was derived from human fetal bone tissue [16]. OPC1 cells were seeded onto the samples placed in 24-well plates. Initial cell density was  $2.0 \times 10^4$  cells  $\text{well}^{-1}$ . A 1 ml aliquot of McCoy's 5A medium (enriched with 5% fetal bovine serum, 5% bovine calf serum and supplemented with  $4 \mu\text{g ml}^{-1}$  of fungizone) was added to each well. Cultures were maintained at  $37^\circ \text{C}$  under an atmosphere of 5%  $\text{CO}_2$ . Medium was changed every 2–3 days for the duration of the experiment. Samples for MTT assay were removed from culture at 3, 7 and 11 days of incubation. The MTT (Sigma, St. Louis, MO) solution of  $5 \text{ mg ml}^{-1}$  was prepared by dissolving 3-(4,5-dimethylthiazole-2-yl)-2,5-diphenyl tetrazolium bromide (MTT) in phosphate-buffered saline and filter sterilizing it. The MTT was diluted ( $50 \mu\text{l}$  into  $450 \mu\text{l}$ ) in serum-free, phenol red-free Dulbecco's minimum essential medium. Then  $500 \mu\text{l}$  of diluted MTT solution was added to each sample in 24-well plates. After 2 h of incubation,  $500 \mu\text{l}$  of solubilization solution made up of 10% Triton X-100, 0.1 N HCl and isopropanol was added to dissolve the formazan crystals. Then  $100 \mu\text{l}$  of the resulting supernatant was transferred into a 96-well plate and read by a plate reader at 570 nm. Data are presented as mean  $\pm$  standard deviation. Statistical analysis was performed using Student's *t*-test and  $p < 0.05$  was considered statistically significant.

### 3. Results and discussion

#### 3.1. Microstructures

Fig. 1 shows the microstructural features of laser processed Ti6Al4V alloy samples with different porosities. Fully dense samples showed characteristic Widmanstätten microstructure and porous Ti alloy samples exhibited more or less equiaxed  $\alpha + \beta$  microstructures. In the Widmanstätten microstructure, the width of  $\alpha$  platelets varied between 2 and  $11 \mu\text{m}$  (average:  $4.6 \pm 2 \mu\text{m}$ ) and the length was in the range of 10 to  $100 \mu\text{m}$  (average:  $38 \pm 19 \mu\text{m}$ ). The average  $\alpha$  phase size was found to be  $9.3 \pm 4.5 \mu\text{m}$  and  $13.4 \pm 5.5 \mu\text{m}$  in the porous Ti alloy samples with 10% and 20% porosity, respectively. The observed variations in microstructural features of laser processed Ti alloy samples with different porosities are attributed to the variations in peak temperatures and cooling rates depending on the extent of powder melting as a result of laser parameters [9]. The Widmanstätten microstructure of fully dense Ti6Al4V alloy sample resulted in relatively high hardness of  $284 \pm 6 \text{ HV}$  than porous Ti alloy samples with equiaxed  $\alpha + \beta$  microstructures,  $251 \pm 11 \text{ HV}$  and  $259 \pm 10 \text{ HV}$  for 10% and 20% porous samples, respectively. Similar higher hardness

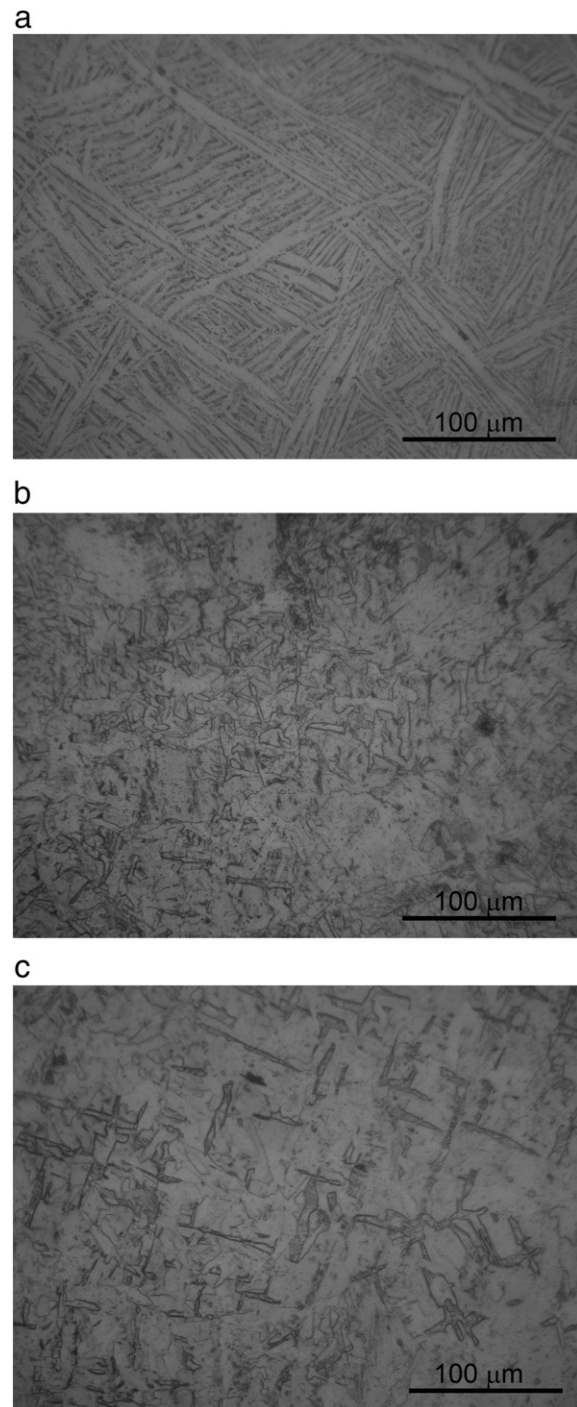


Fig. 1. Microstructures of laser processed Ti6Al4V alloy samples. (a) fully dense sample showing typical Widmanstätten microstructure, (b) sample with 10% porosity, and (c) sample with 20% porosity.

of Widmanstätten microstructures than that of equiaxed microstructures has been reported in wrought Ti6Al4V alloy samples [11] and electron beam rapid manufactured Ti6Al4V alloy [17].

#### 3.2. Mechanical properties

Fig. 2 shows typical shear stress–shear strain curves of laser processed Ti6Al4V alloy samples with different porosity. The torsional yield strength, failure stress and failure strain of present porous Ti6Al4V alloy are significantly higher than that of human cortical bone

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