

ScienceDirect<sup>®</sup>

#### Additive Manufacturing

Volume 41, May 2021, 101916

**Research Paper** 

# Processing, structure, and properties of additively manufactured titanium scaffolds with gyroid-sheet architecture

Cambre N. Kelly <sup>a b</sup> A Kahra <sup>c</sup>, <u>Hans J. Maier</u>, <u>Ken Gall</u> <sup>a b</sup>

Show more 🗸

😪 Share 🍠 Cite

https://doi.org/10.1016/j.addma.2021.101916 ↗ Get rights and content ↗

#### Abstract

While the relationships between processing, structure, and properties of solid titanium alloys produced by <u>additive manufacturing</u> have been established, these relationships are less understood for porous materials, particularly those with rough surfaces inherent to L-PBF. For <u>orthopedics applications</u>, porous architecture and surface roughness are desirable for bone growth, and thus optimization of <u>fatigue life</u> despite these inherent fatigue drivers is critical. The present results establishes relationships between post-processing, microstructure, and resulting fatigue properties for gyroid-sheet scaffolds with as-fabricated surfaces. By comparison of known factors driving <u>fatigue behavior</u>, the relative effect of each on normalized fatigue <u>strength</u> was quantified. Normalized compressive fatigue <u>strength</u> of the gyroid-sheet scaffolds which underwent no surface treatments was observed to be >50%. The result is higher than that seen for tension fatigue of analogous gyroid-sheet scaffolds, or compared to previously reported normalized compressive fatigue strength of strut based scaffolds. The high strength and fatigue resistant behavior of gyroid-sheet scaffolds despite the inherent surface roughness of L-PBF is desirable for <u>biomedical applications</u>.

### Introduction

Additive manufacturing of titanium alloys by laser powder bed fusion (L-PBF) enables fabrication of geometrically complex parts previously unachievable by traditional manufacturing methods. Due to the high spatial resolution of the L-PBF process, architected cellular metals based on complex periodic unit cells

can be designed and manufactured to have prescribed mechanical, mass transport, and biological properties. Recently, sheet-based architectures based on triply periodic minimal surfaces (TPMS) including gyroid, primitive, and diamond-sheet, have gained increasing attention for biomedical, structural, and heat exchange applications. Due to the continuous sheets, these architectures have high surface area, as well as superior strength, energy absorption, and fatigue resistance relative to strut-based architectures of the same volume fraction [1], [2], [3], [4], [5], [6]. Given the gaining momentum for topology optimization and manufacture of TPMS-based scaffolds via L-PBF, processing, structure, property relationships must be developed for this new class of architected porous scaffolds. In this paper, porosity refers to intentionally created free volume in a designed architecture and is distinguished from microvoids, which are unintentional locations of free volume within the solid walls of porous structures.

The mechanical properties of L-PBF samples differ greatly from traditionally wrought or cast ones, including reduced fatigue resistance. Improvement of fatigue life of L-PBF titanium alloy (Ti6Al4V) and other metals may be achieved by altering the printing process itself [7], [8], [9], post-processing heat treatments [10], [11], or surface treatment [12], [13], [14], [15]. Modulation of the printing process includes changing scanning mode (continuous vs. pulsed), scanning strategy, as well as the laser parameters or other machine specific settings [3], [16]. For porous scaffolds, in which strength is significantly lower than for bulk material, optimization of these processing steps is also dependent on the topology of the architecture [17]. As shown recently, modulating energy density in the melt pool significantly improves the monotonic strength and fatigue resistance of gyroid-sheet scaffolds by increasing the material density in solid regions to >99%. The refinement of laser parameters was dependent on the thickness of the sheet walls, as the type of internal defects changed from gas porosity to lack of fusion with increasing wall thickness [3]. Balancing the energy density delivered to the melt pool is imperative in printing of porous structures in order to achieve consolidated material without over melting [7]. Excessive energy density in porous materials results in significant deviation from the designed architecture caused by dross formation or warping of lattice members [3]. Thus, L-PBF process refinement has a limit in improvement of fatigue performance of porous scaffolds once a dense material is achieved, but further improvements can be achieved after the L-PBF process by leveraging post-printing heat treatments.

Due to the high thermal gradients and rapid cooling rates observed in the L-PBF process, the resulting microstructure of as-built titanium alloy parts is a fine lenticular  $\alpha$ ' structure, which can retain significant internal stress [18], [19]. In order to improve ductility and relieve residual stress, post-processing heat treatments are employed. Studies on heat treatments of solid L-PBF titanium alloy both below and above the  $\beta$ -transus have been shown to transform the lenticular structure, promote grain growth, and produce a mixed microstructure leading to improved ductility [11], [12], [14], [15], [19], [20], [21], [22], [23]. Although the temperature, time, and cooling rates of treatments vary, most maximum temperatures are in the range of 800–1050 °C and can be categorized as sub or super-transus. Sub-transus heat treatments (<980 °C) result in an  $\alpha$ -Ti dominated microstructure, reduced residual stress, and grain growth/morphology dependent on treatment temperature and time [10], [11], [22]. In the case of super-transus heat treatments (>980 °C), larger equiaxed grains of  $\alpha$ -Ti with greater presence of  $\beta$ -Ti at the boundaries results in higher ductility. However, this increased grain growth has been associated with more variability in crack growth under cyclic loading, resulting in variable fatigue performance [11]. In both cases, although microstructure transformation and residual stress relief occurs, microvoids within the printed sample can act as crack initiation sites or weak paths for crack propagation during fatigue loading. Hot isostatic pressing (HIP)

2/22/24, 5:50 PM

Processing, structure, and properties of additively manufactured titanium scaffolds with gyroid-sheet architecture - ScienceDirect

treatments performed at sub-transus temperature (895–955 °C) and under high pressure (100MPa) are used for closure of these internal microvoids. In the case of solid titanium alloys with smooth surfaces, crack initiation at internal microvoids was shown to be the primary driver of fatigue failure, while microstructure and residual stress were shown to govern the crack growth phase [11].

In this study, both bulk and scaffold samples were tested without undergoing surface treatment, and thus have surface roughness inherent to the L-PBF process. This inherent roughness can vary from 5 to 20µm for different regions on a given part, and is dependent on the material, printing parameters, as well as orientation of the part relative to the build axis [24], [25]. This surface topography is often reduced by mechanical (milling, blasting, polishing), chemical, or combination treatments. Surface roughness reducing treatments are known to significantly improved the fatigue life of solid titanium alloy [15], [25]. However, for porous materials, mechanical treatments are often limited to exterior surfaces and chemical etching has been shown to have a variable and limited improvement on fatigue life [12], [25], [26]. Further, for biomedical applications, the surface topography is often advantageous for promotion of osseointegration of the implant. Thus, in these cases it is important to improve fatigue resistance through optimization of the scaffold architecture and with other processing parameter, in spite of the surface roughness.

The current study establishes processing, structure, property relationships for solid and porous titanium alloys produced via L-PBF without surface treatment. The overarching goal of the study is to provide a foundational understanding of the effect of thermal post-processing on the tension-tension and compression-compression fatigue properties of L-PBF fabricated materials with as-fabricated surfaces. Although polishing of exposed internal and interconnected porous surfaces is feasible in certain L-PBF applications, some of the most promising applications of printed materials accept and even exploit the natural surface roughness inherent to the printing process. Different than conventional wrought metals and metallic structures, it is important to understand how thermal post-processing and porous structure impact fatigue performance in the presence of these naturally rough and geometrically divergent surfaces. The effect of hot isostatic pressing, sub-transus heat treatment, and super-transus heat treatment are explored and the impact of the various heat treatments on fatigue properties is found to depend on the porous versus solid nature of the material. Further, comparison of tension and compression behavior shows a significant effect of loading mode on the relative fatigue properties of the printed material.

## Section snippets

# Fabrication by laser powder bed fusion

Solid tensile samples (ASTM E8) and porous tensile and compressive samples were fabricated on a 3D Systems ProX DMP 320, using Ti6Al4V ELI (Grade 23) powder under inert argon atmosphere. Powder composition conformed to ASTM F3001, with a particle size distribution of 15–45µm, and spherical morphology. Details of sample geometry and gyroid-sheet design, have been previously described in detail [3]. Briefly, porous compression coupons (cubic) and porous tensile coupons (cubic gage section) were...

# Microstructural analysis

Processing, structure, and properties of additively manufactured titanium scaffolds with gyroid-sheet architecture - ScienceDirect

Microstructure of the titanium alloy produced via L-PBF was shown to be dependent on the post-processing heat treatments applied. The material density was determined via XRM, with the minimum microvoid volume detectable having volume of 8000 µm<sup>3</sup>. In the as-built material, as well at treated at 950 °C and 1050 °C, microvoids were apparent in the volume of the samples, but the material density was > 99% in all despite the persistence of microvoids. In the HIP'ed material, no microvoids with...

## Conclusion

The present results highlight the relationships between post-processing, structure, and fatigue properties in solid and porous titanium alloys deformed under both tension and compression. These results suggest that post-processing approaches to optimize fatigue properties in solid samples do not translate to porous scaffolds where stress concentrations are inherent to the architecture, and a large fraction of untreated surface area is exposed. In these porous scaffolds, there exists a greater...

## CRediT authorship contribution statement

C.N.K., K.G., and H.J.M. were responsible for the experimental design. Execution of the studies was conducted by C.N.K. and C.K. C.N.K. was responsible for writing of the manuscript, and all authors discussed the results and contributed to the review of the final manuscript....

## Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: C.K. and K.G. own stock/stock options in restor3d, Inc. (Durham, NC)....

## Acknowledgements

Financial support from the German Research Foundation (grant MA 1175/67-1) is gratefully acknowledged. The authors also thank J. Baden, A. Krabbenhöft and S. Julmi for help with the microstructural characterization....

Recommended articles

## References (38)

O. Al-Ketan et al.

Topology-mechanical property relationship of 3D printed strut, skeletal, and sheet based periodic metallic cellular materials Addit. Manuf. (2018)

F.S.L. Bobbert et al.

2/22/24, 5:50 PM

Additively manufactured metallic porous biomaterials based on minimal surfaces: a unique combination of topological, mechanical, and mass transport properties Acta Biomater. (2017)

C.N. Kelly et al.

Fatigue behavior of As-built selective laser melted titanium scaffolds with sheet-based gyroid microarchitecture for bone tissue engineering

Acta Biomater. (2019)

H. Gong et al.

Influence of defects on mechanical properties of Ti–6Al–4V components produced by selective laser melting and electron beam melting

Mater. Des. (2015)

L. Thijs et al.

Strong morphological and crystallographic texture and resulting yield strength anisotropy in selective laser melted tantalum

Acta Mater. (2013)

L. Thijs et al.

A study of the microstructural evolution during selective laser melting of Ti–6Al–4V Acta Mater. (2010)

S. Leuders et al.

On the mechanical behaviour of titanium alloy TiAl6V4 manufactured by selective laser melting: fatigue resistance and crack growth performance

Int. J. Fatigue (2013)

S.M. Ahmadi et al.

From microstructural design to surface engineering: a tailored approach for improving fatigue life of additively manufactured meta-biomaterials

Acta Biomater. (2019)

G. Kasperovich et al.

Correlation between porosity and processing parameters in TiAl6V4 produced by selective laser melting

Mater. Des. (2016)

G. Kasperovich et al.

Improvement of fatigue resistance and ductility of TiAl6V4 processed by selective laser melting J. Mater. Process. Technol. (2015)

View more references

https://www.sciencedirect.com/science/article/abs/pii/S2214860421000816?via%3Dihub

Cited by (25)

Influence of mean stress and building orientation on the fatigue properties of sub-unital thinstrut miniaturized Ti6Al4V specimens additively manufactured via Laser-Powder Bed Fusion 2024, International Journal of Fatigue

Show abstract  $\checkmark$ 

Low cycle fatigue behaviour of cellular materials: Experimental comparative study of strutbased and gyroid structures made of additively manufactured 316L steel 2024, International Journal of Fatigue

Show abstract  $\checkmark$ 

Static compression and fatigue behavior of heat-treated selective laser melted titanium alloy (Ti6Al4V) gyroid cylinders

2023, Journal of the Mechanical Behavior of Biomedical Materials

Show abstract  $\checkmark$ 

Prediction of tensile performance for 3D printed photopolymer gyroid lattices using structural porosity, base material properties, and machine learning

2023, Materials and Design

Show abstract  $\checkmark$ 

Selective laser melted Ti6Al4V split-P TPMS lattices for bone tissue engineering

2023, International Journal of Mechanical Sciences

Show abstract  $\checkmark$ 

Microstructure formation mechanisms of spinodal Fe–Cu alloys fabricated using electron-beam powder bed fusion 2023, Journal of Materials Research and Technology Show abstract V



View all citing articles on Scopus 🤇

View full text

© 2021 Elsevier B.V. All rights reserved.



All content on this site: Copyright © 2024 Elsevier B.V., its licensors, and contributors. All rights are reserved, including those for text and data mining, AI training, and similar technologies. For all open access content, the Creative Commons licensing terms apply.

**RELX**<sup>™</sup>