

Short communication: Frequency dependent fatigue behaviour of additively manufactured titanium lattices

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Abstract

Additively manufactured (AM) porous titanium lattices, with their ability to match the mechanical properties of bone and avoid stress shielding, are a popular candidate material for orthopaedic implants. Such implants are now emerging as treatments for conditions like osteoarthritis and fixation of bone fractures. Fatigue tests are critical due to the cyclic loading environment and must be carried out at an accelerated loading rate to simulate many years of use. Tests are typically performed with servohydraulic instruments, which limits the cyclic compression to relatively low frequencies (15 Hz). Fatigue testing at a higher frequency would accelerate research, however, may introduce phenomena such as heat accumulation and strain rate effects. In this study the fatigue behaviour of a pure titanium stochastic lattice was determined at two test frequencies, 15 Hz and 110 Hz. Testing was conducted using an electromechanical dynamic system. The fatigue strengths at 10^6 cycles were 5.607 ± 0.106 MPa and 5.764 ± 0.214 MPa at 15 Hz and 110 Hz respectively. A hypothesis t-test at a 95% confidence level stated that there was significant evidence that the population means were not the same, demonstrating evidence of a difference in fatigue strength with testing frequency. However, we can conclude that the 2.8% increase in fatigue strength due to test frequency effects is inconsequential relative to the time saved (16 hours per test) and typical batch-to-batch variability in fatigue strength of approximately 12%. The results of this study should help accelerate research into the fatigue properties of AM porous lattices.

1 Introduction

Additive manufacturing (AM) of porous metal lattices shows great promise in a wide variety of applications from lightweight aerospace and automotive components, to energy absorbing materials. For medical implants AM enables the matching of material stiffness to that of the surrounding natural bone, minimising stress shielding [1]. These implants are a key treatment for conditions such as osteoarthritis, which affects 344 million people worldwide [2]. Coupled with bone ingrowth into the porous AM lattices, these advances have enormous potential for

improved orthopaedic implants [3]. To date however, the fatigue performance of such lattices is a critical limitation, due to the poor fatigue performance resulting from numerous stress concentrations and surface imperfections left during manufacturing [4]. In a medical setting this is especially relevant due to the highly cyclic nature of the loading on an implant in normal activities such as walking, over several years of use [5]. To simulate the longevity of the implant fatigue tests must be carried out at accelerated loading rates. Usually, testing is conducted at relatively low frequencies (often 15 Hz) due to the limitations of servohydraulic instruments [4], [6] making high cycle fatigue testing ($>10^6$ cycles) excessively time-consuming and infeasible. Modern electromechanical dynamic systems are capable of testing at higher frequencies, however there is a lack of literature on the effects of frequency on the fatigue properties of AM lattices. Fatigue testing standards such as ISO 1099 [7] do not typically require particular test frequencies, but instead recommend selection of a suitable frequency for the particular combination of material, test piece, machine, stiffness, and environmental conditions.

For monolithic (i.e. solid) materials the effects of frequency on fatigue performance have been investigated, for instance by Morrissey et al. [8], [9] on Ti-6Al-4V demonstrating no changes in fatigue strength within the range 60 - 400 Hz, or at 20 kHz. In contrast, pure titanium is observed to display increased fatigue lifetime and endurance limit at 20 kHz compared with 100 Hz [10]. This is influenced by the pure α (HCP – hexagonal close packed) structure of pure titanium, compared with the more complex phase structure of the Ti-6Al-4V alloy, which contains both HCP α and BCC (body-centred cubic) β phases [11]. In contrast to monolithic materials, the frequency dependent fatigue properties of porous materials have rarely been studied. Zettl et al. tested the fatigue behaviour of aluminium foam at 1 - 10 Hz and at 20 kHz, finding no detectable changes in fatigue properties [12]. Despite the increasing prevalence of additively manufactured porous materials over the past decade, to the best of the authors' knowledge no previous works have investigated this effect with additively manufactured lattices. This work presents a brief study of this effect intended to promote further investigation into the underlying high frequency testing phenomena, such as heat accumulation and strain rate effects, and to encourage the use of higher frequencies for longer cycle testing (10^7 cycles, comparable to actual service lifetimes) of medical lattices.

2 Materials & Methods

2.1 Sample Manufacture

Stochastic lattices ($\varnothing 13 \text{ mm} \times 21 \text{ mm}$) with connectivity (number of struts connected to each node) $Z = 12$, strut density $d = 5 \text{ struts/mm}^3$, and strut thickness (diameter) $t = 230 \text{ }\mu\text{m}$ were

designed using Rhinoceros 6 (Robert McNeel & Associates) as described elsewhere [13]. An example is shown in Figure 1. Lattices were fabricated in one batch using an AM250 powder bed fusion system (Renishaw plc.) in commercially pure titanium (grade 2, spherical particle size 15 - 45 μm), using 50 μm layers, 50W laser power and 200-450 μs exposure times. Lattice struts were fabricated using a single laser contour with 70 μm diameter and 50 μm point distance [14], [15]. After fabrication samples were removed from the titanium build plate by electrical discharge machining and ultrasonically cleaned in acetone.

2.2 Quasi-static Testing

In a preliminary test one sample was loaded in compression using a uniaxial testing machine (Instron 5565) at 2 mm/min up to 9 mm. Load and extension data was collected by a 5 kN load cell and two calibrated LVDTs, and an initial estimate of the ultimate strength (σ_U) was obtained from the first peak of the stress-strain curve, before post-yield softening. Stress was defined as the uniaxial force divided by the cross-sectional area of the bulk volume of the lattice sample (13 mm diameter cylinder). Five samples were then loaded to 70% σ_U and reversed to 20% σ_U before loading to failure. The modulus (E) was determined by fitting a linear slope to the hysteresis loop; the yield strength (σ_Y) was then determined at a 1% offset from this (compressive proof strength as per ISO 13314:2011 [16]); and the ultimate strength given by the first peak of the stress-strain curve.

2.3 Fatigue Testing

Compression-compression fatigue testing was carried out using an all-electric dynamic instrument (Instron ElectroPuls E3000) at two test frequencies, 15 Hz and 110 Hz. An Instron dynamic load cell was used, with 5 kN capacity and accuracy of ± 4 N under the maximum loading conditions used (corresponding to ± 0.03 MPa for the samples tested). Testing was carried out in load control using a sinusoidal waveform at the desired frequency, with $R = 0.1$ (minimum/maximum compressive stress ratio); with the test stress set as the maximum stress. The sample and experimental set up are shown in Figure 1. Testing was carried out in accordance with ISO 12107:2012 [17]. This staircase method requires a minimum of 15 valid fatigue tests to accurately estimate the mean and standard deviation of the fatigue strength (σ_f) for a given number of cycles (10^6 cycles). Two additional tests (for a total of 17) were required at 110 Hz to ensure statistical validity of the standard deviation calculation, according to ISO 12107:2012. Testing started at an initial estimate of 22% σ_U (based on previous data) [13] with a step size of 1.25% σ_U [13]. Samples were regarded as failed once a nominal strain of 1% was

exceeded (from crosshead displacement), or when load control could not be achieved due to sample breakage.

Further tests were performed to determine the maximum test frequency for successful load control of 14 different titanium lattice structures (varying Z, d & t to produce structures with varied mechanical properties, as characterised in previous work) [13]. Fatigue testing was conducted at 15% σ_U to 10^5 cycles. If the load was successfully controlled until runout, with the waveform remaining uniform, the test frequency was increased by 5 Hz, otherwise the machine was re-tuned before repeating at the same frequency. This process was repeated until the load could not be controlled regardless of optimisation, or at sample failure. This enables the maximum controllable test frequency to be estimated, for this specific combination of materials, fixtures, and equipment.

3 Results & Discussion

Quasi-static testing (Figure 2) revealed a difference on the order of 15% between the Young's Moduli measured for samples produced in this study, compared with identical structures produced with the same parameters in previous work (Table 1) [13]. However, the relative density (ρ/ρ_s) and ultimate strength were consistent with prior work on these same lattice materials [13]. This difference in modulus can be attributed to the slightly different criteria used to determine the modulus. Previous work [13] conducted a hysteresis loop between 20-70% of the post-yield plateau stress, while this work uses 20-70% of the ultimate stress as used in other previous literature [4], [14], [15]. The ultimate strength, 22.7 ± 0.8 MPa, was used to normalise the fatigue strength at 10^6 cycles to determine the appropriate stress levels for fatigue testing (Figure 3).

The duration of fatigue tests reaching 10^6 cycles at 15 Hz is 18.5 hours, while at 110 Hz this is reduced to 2.5 hours, saving 16 hours per test. This makes high cycle fatigue testing at 10^6 cycles or greater more practical. For instance, at high frequency the high cycle fatigue strength of implants can be determined more easily and quickly, and very high cycle fatigue testing at 10^7 cycles or more is made more feasible. This is critical in encouraging and enabling high cycle fatigue testing to ensure implant safety over the entire lifetime of the device. However it is crucial to be aware of any effect of the testing frequency on the measured fatigue strength. In this work we observe that increasing the test frequency from 15 to 110 Hz increases the mean fatigue strength by 2.8% from 5.607 ± 0.106 to 5.764 ± 0.214 MPa (Table 1). The statistical significance of this effect was estimated using standard methods [17], [18]. A two-tailed hypothesis t-test was carried out at a 95% confidence level, with the null hypothesis that the population means of the two groups are equal. The corresponding p-value = 0.015, therefore

the population means cannot be said to be equal. The fatigue strength at 110 Hz is higher, demonstrating some evidence of test frequency effects. Further research could be carried out to determine the physical basis for this difference, investigating whether other high frequency fatigue testing phenomena such as heat accumulation occur by using optical thermometry. Strain rate hardening behaviour such as this is typical for metals deforming by dislocation slip, including CP-Ti with its HCP crystal structure, where there is reduced time for dislocations to overcome barriers through thermal activation [19], [20].

The frequency dependence of fatigue behaviour in metals is generally considered to be influenced by three main factors; strain rate, environment, and specimen heating [11]. Specimen self-heating occurs as a result of energy dissipation from inelastic deformation caused by dislocation movement, and becomes more significant at higher stresses. Local heating can activate additional dislocation movement resulting in reduced strength [21], [22]. These effects are expected to be small in this work, because of the modest test frequencies of 15-110 Hz used, compared with the ultrasonic (20 kHz) frequencies often used in thermal dissipation studies. The highly porous (~80% porosity) nature of these samples will also serve to prevent any thermal buildup and lessen heating effects. Environmental interactions are also known to play a role in the frequency dependence of fatigue behaviour, as the surface oxide layer that forms in corrosive environments (air, humid air) can be carried beneath the surface and increase the fatigue damage, increasing crack growth rates [11], [23]. The time dependence of the mass transport and reaction processes can lead to frequency dependent fatigue behaviour, which can be suppressed in some materials by testing in an inert atmosphere [24]. Such behaviour has been documented in several Al alloys, but the frequency dependence of titanium fatigue is not known to show strong dependence on the environment. Nevertheless environmental effects should be carefully considered, especially for biomedical applications where combined fatigue-corrosion tests may be necessary – acceleration of such tests to high frequency should be conducted with caution.

For pure titanium such as used here, strain rate effects are likely to be the dominant factor controlling the frequency dependence of fatigue behaviour. The results observed here – a modest increase in fatigue strength at higher frequency – are consistent with results from Papakyriacou on annealed CP-Ti [10]. This is a result of the known strain rate dependent behaviour of pure titanium as demonstrated by Sakamoto et al. due to visco-plastic strain behaviour at the crack tip, affecting both crack initiation life and crack propagation rate [25]–[27]. The HCP crystal structure of CP-Ti plays a key role here, with the dominant prismatic and basal slip systems both demonstrated to show strain rate dependence [28]. There are further

factors at play during AM of CP-Ti that can potentially influence the strain rate dependence. Metal AM is known to produce particular crystallographic textures – for Ti alloys columnar β grains form with preferential orientation of $\langle 001 \rangle_\beta$ directions parallel to the build direction, leading to orientation of the α/α' phase with the c-axis at $\sim 30\text{-}45^\circ$ to the build direction [29]–[31]. Such crystallographic texture impacts the strain rate sensitivity of the material as shown by Lee et al., depending on the relative orientation of the more strain rate sensitive slip system (basal) with respect to the loading direction [28], [32]. The crystallographic texture resulting from laser powder bed fusion of CP-Ti results in a large proportion of grains oriented for easy basal slip [31], which can explain the strain rate dependence seen here. The AM process is also known to result in oxygen enrichment, leading to strengthening and reduced ductility [33], [34]. Such increases in oxygen solute content have recently been shown to reduce the strain rate sensitivity [32], which may result in AM CP-Ti being less strain rate sensitive than wrought CP-Ti, however more research is needed to compare the magnitude of this effect to other AM effects such as crystallographic texture. AM also enables complex structures such as foams and lattices to be produced, whose frequency dependent fatigue behaviour has rarely been studied. Zettl et al. found no effect of frequency for Al alloy foams [12] – although FCC metals have low strain rate sensitivity, environmental effects can be significant for Al alloys, however they were not observed in this study conducted in ambient air. In this work the strain rate dependence of porous CP-Ti appears consistent with literature on monolithic CP-Ti, however further work is needed to separate the effects of AM and the porous structure on fatigue frequency sensitivity.

Despite this measurable difference in normalised high cycle fatigue strength between tests at 15 Hz and 110 Hz, these results must be considered in context of other sources of variation such as the batch-to-batch variability often seen in additive manufacturing. Comparing these results to previous work on the same structures [13], the batch-to-batch difference between the normalised fatigue strength at 10^6 cycles at 15 Hz is appreciably larger at 11.6% than the frequency effect observed here. This variation between batches can be partly attributed to the metal powder properties, which are beginning to be considered more carefully in recent years [35]. The recycling and reuse of expensive metal powder between batches can lead to changes in powder properties, including chemical composition, porosity, morphology, and flowability, in turn affecting the mechanical properties, in particular surface-sensitive fatigue processes. In addition, failure by fatigue is an inherently stochastic process with a significant amount of scatter as demonstrated by the measured standard deviation, making the observed difference due to test frequency negligible in comparison. Relative to this variation observed for the additive manufacturing and fatigue testing processes, the 2.8% difference due to test frequency

effects during higher frequency testing can be considered inconsequential compared with the time saved.

There is a positive relationship between the maximum test frequency for successful load control and the mechanical properties (E , ρ/ρ_s and σ_U) of a stochastic titanium lattice structure (Figure 4). These results provide a reference for estimation of the achievable test frequency for materials with known mechanical properties, to facilitate experimental planning and optimisation. The physical basis for this limitation in frequency is likely the power available for the linear actuators, which is used to accelerate and decelerate the test fixtures at the speed required. Materials with lower stiffness will require larger displacements to reach the same compressive load as materials with higher stiffness, requiring a higher maximum speed for the actuator head even at a constant frequency. As a result, the maximum achievable frequency tends to be lower for materials with lower stiffness. The material properties are however not the only determining factor affecting the maximum test frequency achievable. The load string stiffness (LSS), a measure of the compliance of the entire load assembly including the sample and test fixtures, plays a key role. In this work the load string stiffness and sample modulus are highly correlated (Figure 4), as different lattice materials are tested with the same test fixtures. However any changes to the test setup, for instance different compression fixtures, would be expected to alter the load string stiffness and therefore change the maximum possible test frequency.

4 Conclusions

This study investigated the frequency dependent fatigue behaviour of AM titanium lattices. Two sample sets were tested at 15 Hz and 110 Hz. It was found that the Z12-d5 ($t=230\mu\text{m}$) structure had a fatigue strength at 10^6 cycles of 5.607 ± 1.06 MPa at 15 Hz and 5.764 ± 0.214 MPa at 110 Hz. There is sufficient evidence to conclude from a hypothesis t-test (95% confidence) that population means are not equal and thus, the increase in fatigue strength is due to test frequency effects. This 2.8% difference is however inconsequential relative to the batch-to-batch variation in fatigue strength of approximately 12% and the time saved (16 hours per test). These results should encourage the use of higher loading frequencies for fatigue tests of AM lattice structures, accelerating research and enabling more load cycles during testing, more accurately reflecting real-world service conditions.

Credit authorship contribution statement

A.C. Jones: Methodology, Formal analysis, Investigation, Visualisation, Writing – Original Draft. **J.R.T. Jeffers:** Writing – review & editing, Supervision, Funding acquisition. **R.N.**

Oosterbeek: Conceptualisation, Methodology, Writing – review & editing, Supervision, Funding acquisition.

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Table 1: Mechanical properties (n=5) and fatigue behaviour of pure titanium Z12-d5 stochastic lattice structure (t=230 μ m) of two separate batches, presented as mean $\pm$ standard deviation [13]. The fatigue strength is normalised as σ_f/σ_U .

	This work		Prior work [13]
E (MPa)	2672.3 $\pm$ 117.2		2268.2 $\pm$ 121.0
ρ/ρ_s (%)	19.1 $\pm$ 0.5		19.3 $\pm$ 0.4
σ_U (MPa)	22.7 $\pm$ 0.8		22.4 $\pm$ 0.6
Test frequency (Hz)	110	15	15
σ_f at 10⁶ cycles (MPa)	5.764 $\pm$ 0.214	5.607 $\pm$ 0.106	4.91 $\pm$ 0.61
Normalised σ_f at 10⁶ cycles	0.254 $\pm$ 0.009	0.247 $\pm$ 0.005	0.22 $\pm$ 0.03

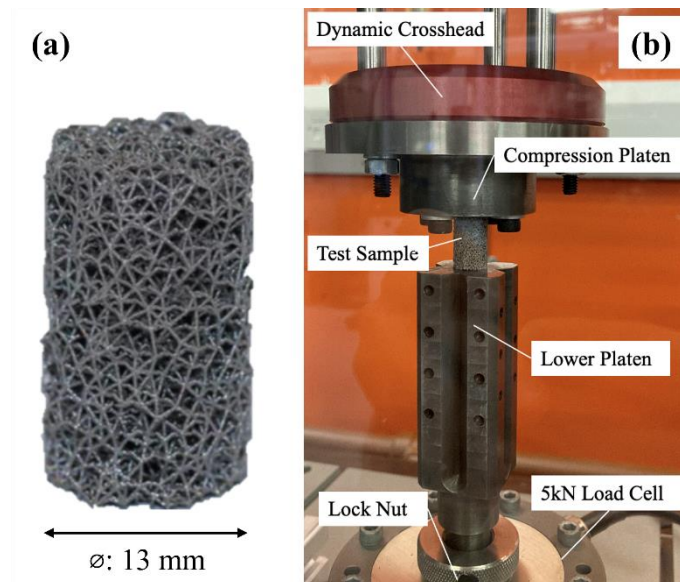


Figure 1: Photographs showing (a) additively manufactured stochastic porous titanium lattice sample, and (b) experimental setup for compression-compression fatigue testing in Instron ElectroPuls E3000 system.

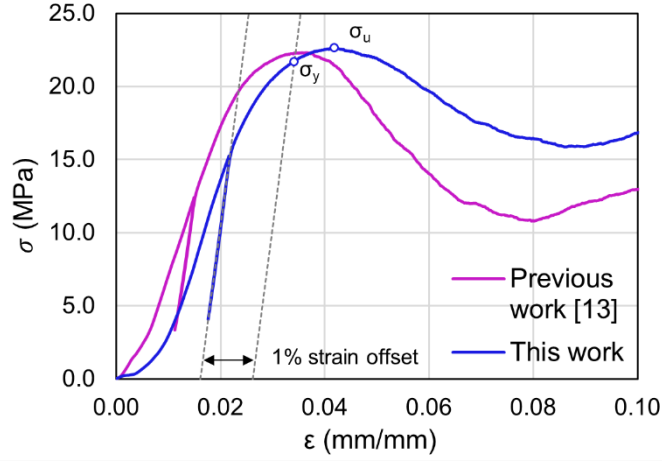


Figure 2: Compressive stress-strain curves for the stochastic lattices used in this study (CP-Ti, $Z=12$, $d=5$, $t=230\text{ }\mu\text{m}$), compared with equivalent data from previous work [13].

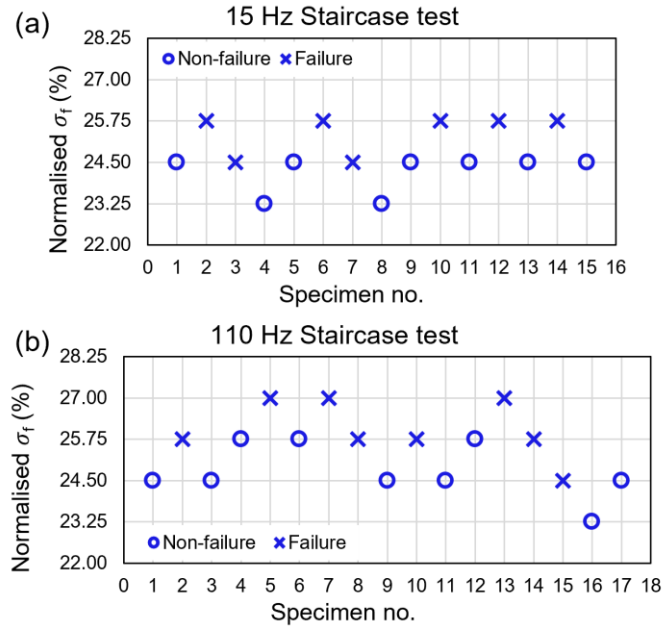


Figure 3: Fatigue test results from staircase tests of additively manufactured stochastic porous titanium lattices at 15 Hz (a) and 110 Hz (b), showing the test stress normalised to the quasi-static ultimate strength (σ_U).

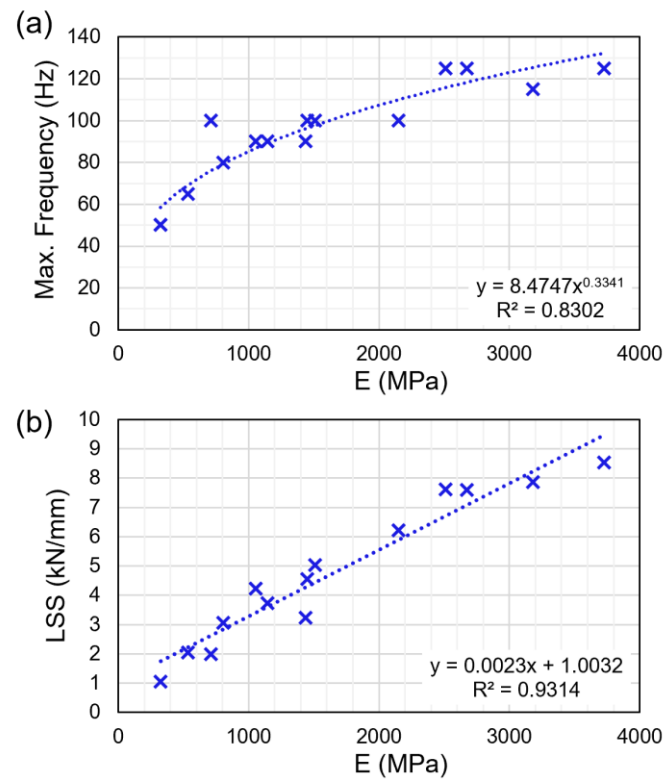


Figure 4: Results from tests of maximum controllable frequency for additively manufactured stochastic porous titanium lattices with different mechanical properties. Maximum test frequency shown plotted against sample elastic modulus (a), and the dependence of load string stiffness (LSS) on sample elastic modulus (b).