Frequency dependent fatigue behaviour of additively manufactured titanium lattices

Adam C. Jones[1], Jonathan R.T. Jeffers[1], Reece N. Oosterbeek[1,2,*]

[1] Department of Mechanical Engineering, Imperial College London, London, SW7 2AZ, United Kingdom

[2] Department of Engineering Science, University of Oxford, Parks Road, Oxford OX1 3PJ, United Kingdom

* Corresponding Author: R N Oosterbeek (reece.oosterbeek@eng.ox.ac.uk)

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. These implants are a key treatment for conditions such as osteoarthritis, which affects 344 million people worldwide [1]. 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 cyclic hardening. In this study the fatigue behaviour of a pure titanium Z12-d5 stochastic lattice (t = 230 μm) 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 cyclic hardening. However, we can conclude that the 2.8% increase in fatigue strength due to cyclic hardening 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 [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 Guennec et al., finding that the fatigue strength of low carbon steel increased when the test frequency increased from 0.2 to 140 Hz [8]. This behaviour is termed cyclic hardening. This effect is also seen to depend on the material, with work by Morrissey et al. [9], [10] on Ti-6Al-4V (a similar material to that used in this study) demonstrating no changes in fatigue strength within the range 60 - 400 Hz, or at 20 kHz. 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 [11]. 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 cyclic hardening or softening, 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 (Ø13 mm × 21 mm) with connectivity Z = 12, strut density d = 5 struts/mm^3, and strut thickness t = 230 µm (Figure 1) were designed using Rhinoceros 6 (Robert McNeel & Associates) as described elsewhere [12]. 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 [13]. 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. 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; 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. The sample and experimental set up are shown in Figure 1. Testing was carried out in accordance with ISO 12107:2012 [14]. 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) [12] with a step size of 1.25% σ_U [12]. 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) [12]. 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 revealed a batch-to-batch 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) [12]. However, the relative density (ρ/ρ_s) and ultimate strength were consistent with prior work on these same lattice materials [12]. This variation between batches can be attributed to the metal powder properties, which are beginning to be considered more carefully in recent years [15]. 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. 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 2).

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 [14], [16]. 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 cyclic hardening. 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.

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 [12], 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. Relative to this variation observed for the additive manufacturing and fatigue testing processes, the 2.8% difference due to cyclic hardening 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 3). 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 3), 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=230μm) 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 cyclic hardening. 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.

Acknowledgments

The authors wish to gratefully acknowledge support from the Imperial College London Mechanical Engineering UROP Bursary, the National Institute of Health Research (NIHR300013), and the Engineering and Physical Sciences Research Council (EP/R042721/1).

References

[1] A. Cieza, K. Causey, K. Kamenov, S. W. Hanson, S. Chatterji, and T. Vos, "Global estimates of the need for rehabilitation based on the Global Burden of Disease study 2019: a systematic analysis for the Global Burden of Disease Study 2019," *Lancet*, vol. 396, no. 10267, pp. 2006–2017, Dec. 2020, doi: 10.1016/S0140-6736(20)32340-0.

[2] D. R. Sumner, "Long-term implant fixation and stress-shielding in total hip replacement," *J. Biomech.*, vol. 48, no. 5, pp. 797–800, Mar. 2015, doi: 10.1016/J.JBIOMECH.2014.12.021.

[3] N. Koju, S. Niraula, and B. Fotovvati, "Additively Manufactured Porous Ti6Al4V for Bone Implants: A Review," *Met. 2022, Vol. 12, Page 687*, vol. 12, no. 4, p. 687, Apr. 2022, doi: 10.3390/MET12040687.

[4] S. Ghouse, R. N. Oosterbeek, A. T. Mehmood, F. Vecchiato, D. Dye, and J. R. T. Jeffers, "Vacuum Heat Treatments of Titanium Porous Structures," *Addit. Manuf.*, vol. 47, p. 102262, 2021, doi: 10.1016/j.addma.2021.102262.

[5] G. Bergmann *et al.*, "Hip contact forces and gait patterns from routine activities," *J. Biomech.*, vol. 34, no. 7, pp. 859–871, Jul. 2001, doi: 10.1016/S0021-9290(01)00040-9.

[6] H. M. A. Kolken, A. F. Garcia, A. Du Plessis, C. Rans, M. J. Mirzaali, and A. A. Zadpoor, "Fatigue performance of auxetic meta-biomaterials," *Acta Biomater.*, Mar. 2021, doi: 10.1016/j.actbio.2021.03.015.

[7] International Organization for Standardization, *ISO 1099:2017 Metallic materials — Fatigue testing — Axial force-controlled method*. BSI Standards Limited, 2017.

[8] B. Guennec, A. Ueno, T. Sakai, M. Takanashi, Y. Itabashi, and M. Ota, "Effect of the loading frequency on fatigue properties of JIS S15C low carbon steel and some discussions based on micro-plasticity behavior," *Int. J. Fatigue*, vol. 70, pp. 328–341, 2015, doi: 10.1016/j.ijfatigue.2014.10.006.

[9] R. J. Morrissey and T. Nicholas, "Fatigue strength of Ti–6Al–4V at very long lives,"

Int. J. Fatigue, vol. 27, no. 10–12, pp. 1608–1612, Oct. 2005, doi: 10.1016/J.IJFATIGUE.2005.07.009.

[10] R. J. Morrissey and P. J. Golden, "Ultrasonic fatigue testing of Ti-6Al-4V," *J. ASTM Int.*, vol. 2, no. 5, 2005, doi: 10.1520/stp11501s.

[11] B. Zettl, H. Mayer, and S. E. Stanzl-Tschegg, "Fatigue properties of Al-1 Mg-0.6 Si foam at low and ultrasonic frequencies," *Int. J. Fatigue*, vol. 23, no. 7, pp. 565–573, 2001, doi: 10.1016/S0142-1123(01)00025-1.

[12] S. Kechagias, R. N. Oosterbeek, M. J. Munford, S. Ghouse, and J. R. T. Jeffers, "Controlling the mechanical behaviour of stochastic lattice structures: The key role of nodal connectivity," *Addit. Manuf.*, vol. 54, p. 102730, Jun. 2022, doi: 10.1016/J.ADDMA.2022.102730.

[13] S. Ghouse, S. Babu, K. Nai, P. A. Hooper, and J. R. T. Jeffers, "The influence of laser parameters, scanning strategies and material on the fatigue strength of a stochastic porous structure," *Addit. Manuf.*, vol. 22, pp. 290–301, Aug. 2018, doi: 10.1016/j.addma.2018.05.024.

[14] International Organization for Standardization, *ISO 12107:2012 Metallic materials — Fatigue testing — Statistical planning and analysis of data*. Internation Organization for Standardization, 2012.

[15] P. Moghimian *et al.*, "Metal powders in additive manufacturing: A review on reusability and recyclability of common titanium, nickel and aluminum alloys," *Addit. Manuf.*, vol. 43, Jul. 2021, doi: 10.1016/J.ADDMA.2021.102017.

[16] W. J. Dixon and A. M. Mood, "A Method for Obtaining and Analyzing Sensitivity Data," *J. Am. Stat. Assoc.*, vol. 43, no. 241, pp. 109–126, 1948, doi: 10.1080/01621459.1948.10483254.

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

	This work		**Prior work[5]**
E (MPa)	2672.3 ± 117.2		2268.2 ± 121.0
ρ/ρ_s (%)	19.1 ± 0.5		19.3 ± 0.4
σ_U (MPa)	22.7 ± 0.8		22.4 ± 0.6
Test frequency (Hz)	**110**	**15**	**15**
σ_f at 10^6 cycles (MPa)	5.764 ± 0.214	5.607 ± 0.106	4.91 ± 0.61
Normalised σ_f at 10^6 cycles	0.254 ± 0.009	0.247 ± 0.005	0.22 ± 0.03

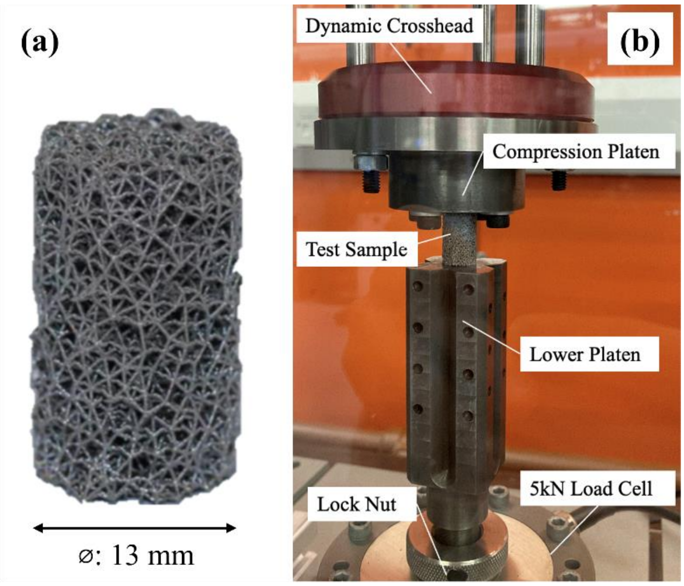

Figure 1: Photographs showing (a) additively manufactured stochastic porous titanium lattice sample, and (b) experimental setup for compression-compression fatigue testing in Instron ElectroPuls E3000 system.

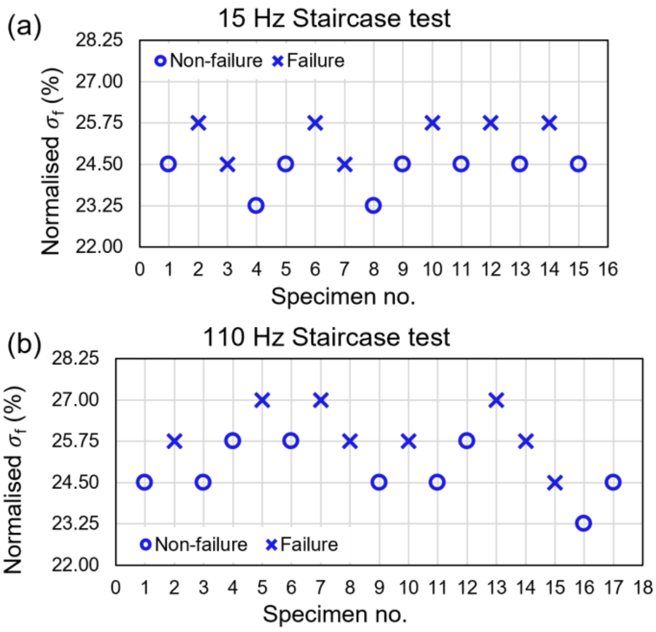

Figure 2: Fatigue test results from staircase tests of additively manufactured stochastic porous titanium lattices at 15 Hz (a) and 110 Hz (b), showing the test stress normalised to the quasi-static ultimate strength (σ_U).

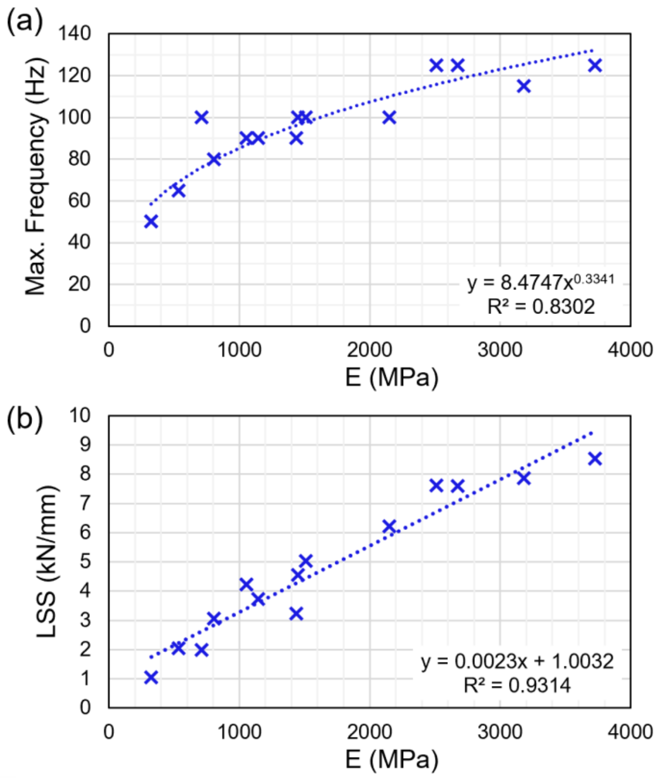

Figure 3: Results from tests of maximum controllable frequency for additively manufactured stochastic porous titanium lattices with different mechanical properties. Maximum test frequency shown plotted against sample elastic modulus (a), and the dependence of load string stiffness (LSS) on sample elastic modulus (b).